

[19] Peoples' Republic of China Patent Office

[51] Int. Cl⁶

C09J133/08

C09J 7/02

[12] Patent Application Public Announcement

[21] Application No. 95192420.6

[43] Public Announcement Date: March 12, 1997

[11] Public Announcement No. CN 1145087A

[22] Date of Application: February 24, 1995

[30] Priority

[32] April 4, 1994 [33] US [31] 08/222,458

[86] International Application: PCT/US95/02295 February 24, 1995

[87] International Announcement: WO95/27016 Great Britain October 12, 1995

[85] Date Advanced to National Phase: October 3, 1996

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Patent claims 3 pages; descriptions 24 pages; attached figures 0 page.

[54] Name of Invention: Pressure Sensitive Adhesive and Its Related Products

[57] Abstracts

This invention provides a water dispersible pressure sensitive adhesive. In one application example, the said adhesive consists of micro (granular) particles and a water dispersible component. In another application example, the said adhesive consists of a novel water dispersible polymer with a large amount of polyalkoxy-alkyl-acrylate as its major component. The said polymer may be used as the water dispersion component. The pressure sensitive adhesive can be prepared into tapes, and used as label for container, sterilization indicator and label, sealing system for envelope, surgical dressing (bandage), and body cover for mammals. It can also be used to prepare a splicing material for paper rolls; in another application example, the adhesive tape is repulpable; yet in another application example, it is washable.

Patent Claims

1. An usually tacky (sticky) pressure sensitive adhesive composition, which consists of a mixture of the following components:

- A) A solvent insoluble but dispersible elastomeric polymer micro-particles component; and
- B) a water dispersible polymer component.

2. A pressure sensitive adhesive composition as in Claim 1, the characteristics of which is that it consists of 0.1-99.9 parts of the said micro particle component by weight, and corresponding 99.1-0.1 parts by weight of the said water dispersible component.

3. A pressure sensitive adhesive composition as in Claim 1, the characteristics of which is that portion of the micro particle component may be solid particles, or particles containing at least one void.

4. A pressure sensitive adhesive composition as in Claim 3, the characteristics of which is that:

(A) The micro particle component is consisting of polymer with the following components:

(1) 100 parts or less by weight of at least one type of free radical polymerizable monomer, the said monomer is selected from alkyl acrylate, alkyl methacrylate, vinyl ester and their mixtures;

(2) 0-30 parts by weight of at least one polar monomer that is capable of copolymerize with the free radical polymer monomer A (1);

(3) 0-40 parts by weight of at least of a hydrophilic oligomer or polymer component that is capable of copolymerizing with the free radical polymer monomer A (1) or the polar monomer A (2);

(B) The water dispersible polymer component consists of polymers with the following components:

(1) 50-98 parts by weight of a water soluble poly-alkoxy alkyl acrylate, and

(2) 50-2 parts by weight of a carboxylic acid.

5. A pressure sensitive adhesive composition as in Claim 4, the characteristics of which is that the micro particle component consists of 80-98 parts (by weight) of a free radical polymerizable monomer, 1-17 parts by weight of a polar monomer, and 1-18 parts by weight of a hydrophilic oligomer or polymer.

6. A pressure sensitive adhesive composition as in Claim 5, the characteristics of which is that the said hydrophilic component consists of macro monomers with the following general formula:



where: X is a functional group that is copolymerizable with a free radical polymerizable monomer and an optional polar monomer;

Y is a divalent linking group;

n is an integer 0 or 1; and

Z is a mono-valent hydrophilic polymer or oligomer with a degree of polymerization equal to or greater than 2.

7. A pressure sensitive adhesive composition as in Claim 5, the characteristics of which is that the water dispersible polymer component also consists of 0-40 parts by weight of an essentially water insoluble alkyl acrylate and 0-6 parts by weight of an emulsifier monomer.

8. A pressure sensitive adhesive composition as in Claim 7, the characteristics of which is that (it consists of) 80-95 parts by weight of a water soluble polyakloxy alkyl acrylate, 20-5 parts by weight of the said carboxylic acid, and 0-4 parts by weight of the said emulsifier monomer.

9. A pressure sensitive adhesive sheet, which consists of a layer of an usually tacky (sticky) pressure sensitive adhesive as described in Claim 1.

10. A pressure sensitive adhesive tape as in Claim 9, the characteristics of which is that the said usually tacky pressure sensitive adhesive sheet is coated on at least one side of a flexible (soft) carrier (support) material.

11. A pressure sensitive adhesive tape as in Claim 10, the characteristics of which is that the said usually tacky pressure sensitive adhesive layer is removably bonded to the said flexible support.

12. A pressure sensitive adhesive tape as in Claim 11, the characteristics of which is that the said usually tacky pressure sensitive adhesive layer is essentially permanently bonded to the said flexible support.

13. A pressure sensitive adhesive tape as in Claim 12, the characteristics of which is that the said tape is repulpable.

14. A repulpable pressure sensitive adhesive tape, consists of a thin layer of an usually tacky pressure sensitive adhesive composition as in Claim 1, the characteristics of which is that the micro particle component is a suspension polymerization material.

15. A water dispersible pressure sensitive adhesive tape as in Claim 14, the characteristics of which is that it consists of 10-95 parts by weight of the said micro particle component, and 90-5 parts by weight of the said water dispersible polymer component.

16. A repulpable pressure sensitive adhesive tape, , as in Claim 15, the characteristics of which is that it consists of 50-95 parts by weight of the said micro particle component, and 50-5 parts by weight of the said water dispersible polymer component.

17. A washable pressure sensitive adhesive tape, as in Claim 15, the characteristics of which is that it consists of 10-50 parts by weight of the said micro particle component, and 90-50 parts by weight of the said water dispersible polymer component.

18. A washable pressure sensitive adhesive tape, as in Claim 17; the characteristics of which is that it consists of 10-40 parts by weight of the said micro particle component, and 90-60 parts by weight of the said water dispersible polymer component.

19. A pressure sensitive adhesive tape as in Claim 14, the characteristics of which is that the said micro particle component is a pressure sensitive adhesive.

20. A pressure sensitive adhesive tape as in Claim 14, the characteristics of which is that the said water dispersible polymer component is a pressure sensitive adhesive.

21. A fabric that is covered with the pressure sensitive adhesive tape described in Claim 18.

22. A fabric as in Claim 21, the characteristics of which is that it is in the form of a cover for the body of a mammal.

23. A pressure sensitive adhesive tape as in Claim 10, the characteristics of which is that it is in the form of a sterilization indicator.

24. A splicing material, that consists of pressure sensitive adhesive tape described in Claim 14, for joining the first and the second re-pulp able tapes.

25. A water dispersible pressure sensitive adhesive polymer that is used as component B) of Claim 1 it consists of emulsion polymerization products with the following compositions:

- A) 50-98 parts by weight of a poly alkoxy alkyl acrylate, and
- B) 50-2 parts by weight of a carboxylic acid;
- C) 0-40 parts by weight of an essentially water insoluble alkyl acrylate;
- D) 0-6 parts by weight of an emulsion monomer that is copolymerizable with the said poly alkoxy alkyl acrylate, or the carboxylic acid.

26. A pressure sensitive adhesive tape, the characteristics of which are that it includes a layer of an usually tacky pressure sensitive adhesive polymer as in Claim 25.

27. A pressure sensitive adhesive tape in Claim 26, the characteristics of which is that the said pressure sensitive adhesive polymer layer is coated, at least partially, on some area of at least one surface of a flexible carrier material.

28. A pressure sensitive adhesive tape, which includes a flexible repulp-able carrier layer, at least portion of the first surface of the said carrier has an usually tacky pressure sensitive adhesive polymer layer as described in Claim 25; at least portion of the second surface of the said carrier layer has a release agent; the said second surface of the said carrier layer is on the opposite side of first surface of the said carrier.

Descriptions

Pressure Sensitive Adhesive and Its Related Products

Technical Domain of the Invention

This invention concerns a pressure sensitive adhesive and its related products.

Background of Invention

Pressure sensitive adhesive (PSA) has been widely used in many applications. The characteristics of these adhesives are that they are usually tacky (sticky) at room temperature (such as 20°C); at most, it requires only a very light finger pressure to bond to a surface. They exhibit a balance between the viscoelasticity and elasticity; such balance leads to the quadruple balance among adhesion, cohesion, tensile property, and elasticity. They have sufficient cohesiveness and elasticity, thus they can be bonded through touching and also be removed from a surface, without leaving a residue even if it is very tacky. Pressure sensitive adhesive should not contain composition merely because such composition is tacky or capable of bonding to a surface.

Pressure sensitive adhesive containing micro particles is one type of pressure sensitive adhesive. Such pressure sensitive adhesive contains a large amount of micro particles; these micro particles may be, or may be not an inherently pressure sensitive adhesive; they may be hollow, or solid particles. Micro particle containing pressure sensitive adhesive may also contain bonding agent that can help to secure the micro particles to a substrate. If necessary, such adhesive may be inherently pressure sensitive; for example, refer to U.S. 3,857,731 (Merril et al.); U.S. 4656,218 (Kinoshita); U.S. 4,735,837 (Miyasaka et al.); U.S. 4,855, 170 (Durvall et al.); U.S. 4,994,322 (Delgado et al.); U.S. 5,118,750 (Silver et al.); and WO 93/02855 (Mallya et al.). The adhesive made public in these literatures are not water dispersible. When re-pulping, they form clusters, and producing adhesive globlets. These globlets (refer to as "adhesive material" (?) in the field) will clog up the sieve used in the re-pulping process, resulting in an increase of production time and cost. In addition, any globe that can not be removed by the sieve will affect the quality of the recycled paper produced. Therefore, there is a demand for readily repulpable water dispersible pressure sensitive micro particle adhesive.

Brief Description of Invention

This invention overcomes the disadvantages of present technology. On one hand, this invention is providing an usually tacky pressure sensitive adhesive composition, which is composed of the following mixtures:

- A) A polymer micro particle component which is not soluble but dispersible in a solvent; and
- B) a water dispersible polymer component.

The micro particle component consists of 0.1-99.9 parts by weight of the adhesive composition, and correspondingly, 99.9-0.1 parts by weight of the water dispersible adhesive composition.

On the other hand, this invention provides a water dispersible pressure sensitive adhesive

composition, the said composition consists of the following emulsion polymerization products:

A) Approximately 50-98 parts by weight, but preferably approximately 80-95 parts by weight of poly alkoxy alkyl acrylate, and

B) approximately 50-2 parts by weight, but preferably approximately 20-5 parts by weight of a carboxylic acid.

These polymers, consist of primarily poly alkoxy alkyl acrylate elements, are pressure sensitive adhesive.

The adhesive composition of this invention is preferably water dispersible. Comparing to the current technology, it has one or more improvements in the performance of adhesion, tackiness, and moisture resistance. Such adhesive composition may be prepared into a bulk form or any necessary pressure sensitive adhesive sheets of required width. It could be a narrow strip, a tape, or a wide film. In all cases, the pressure sensitive adhesive sheet is a thin layer (in general, the thickness is less than 125 μ). In addition, the adhesive layer may be coated on one or more corresponding surfaces of a flexible substrate; if the adhesive layer is removable from the coated substrate, the product is referred to as an adhesive transfer tape; if the adhesive is essentially permanently bonded to the substrate, then the carrier is referred to as a backing.

The adhesive thin film of this invention has many applications. For example, it can be used to manufacture repulpable paper splicing material (permanent or temporary). In addition, such adhesive may be used in label for container, sterilization indicator tape and label, sealing system for container such as envelope, sealing system for surgical dressing, and body cover for mammals (such as surgical gown, sheet, and drape, etc.). After reading this disclosure, other applications for the adhesive and tape of this invention become apparent to those technical personnel who are familiar with the field.

In this description, the following terms have the following meanings:

“Water dispersible” refers to those particles that pass TAPPI UM-213 Test or Washing Test. The two tests mentioned above will be described in details later.

“Mixing” is referring to the physical mixing of the micro particles and water dispersible polymer component. Although they may be binding through hydrogen bonds, but these components are not covalently bonded.

Detailed Descriptions

Micro Granular Particle Component

The micro particle component used in this invention consists of polymeric elastomeric micro particles that are insoluble in a solvent but is dispersible in the solvent. If necessary, these micro particles may be an inherently pressure sensitive adhesive, although it is not required. In addition, if necessary, a crosslinking agent may be added. During preparation, the micro particles are usually spherical. Their average diameter is usually 1-300 microns (preferably 1-50 microns). The micro particles used in this invention may be solid, or hollow. Hollow micro particles may have more than one pore. In general, the average diameter of the pores of the micro particles is less than 100 microns.

The micro particles may be prepared through various emulsification processes. For example, the micro particles may be prepared by suspension polymerization with the following “two step”

emulsion polymerization process. The process includes the following steps:

a) Formation

(i) Mix an aqueous solution of a polar monomer with an oil phase monomer to form a water-in-oil emulsion; the oil phase monomer is selected from alkyl (meth)acrylates and vinyl acrylates;

(ii) the water-in-oil emulsion is dispersed in an aqueous phase, to form a water-in-oil-in-water emulsion;

b) Initiate polymerization, preferably through heating or radiation.

In the said method, the first step preferably involves formation of a water-in-oil emulsion of an aqueous solution monomer (at least portion of which is a polar monomer) in an oil phase monomer (namely, at least one (meth) acrylate or vinyl ester monomer, and optional hydrophilic oligomer and/or polymer with free radical reactivity), with an emulsify agent with a low hydrophilic-lyophilic-balance (HLB) value. A suitable emulsifier has a HLB value of approximately smaller than 7, preferably 2-7; examples of such emulsifiers include sorbitan mono-oleate, sorbitan tri-oleate, and ethoxylated oleyl alcohol such as BrijTM 93 (obtained from Atlas Chemical Industries, Inc.).

In the first phase of the first step, an oil phase monomer, an emulsifier, a free radical initiator, and an optional free radical reactive hydrophilic oligomer and/or polymer, as well as optional cross-linked monomer or monomer defined below are combined; stir the aqueous solution containing all or portion of the polar monomer(s) then pour into the oil phase mixture to form a water-in-oil emulsion. Hydrophilic oligomer and/or polymer with free radical reactivity may be added to the oil phase or aqueous phase. Thickening agent, such as methyl cellulose, may be added to the water phase of the water-in-oil emulsion. In the second phase of the first step, the water-in-oil emulsion is dispersed in the aqueous phase of an emulsifier with a HLB value of approximately 6, forming a water-in-oil-in-water emulsion. The aqueous phase may also contain the optional portion of the polar monomer(s) that is not added in the first step. The examples of these emulsifiers include ethoxylated sorbitan mono-oleate, ethoxylated lauryl alcohol and alkyl sulfate. In both steps, when using an emulsifier, its concentration should be larger than its critical micelle concentration; the critical micelle concentration is defined in this article as the minimum emulsifier concentration required to form micelles (submicroscopic aggregates of emulsifier molecules). The critical micelle concentration is slightly different for each emulsifier; the useful concentration is approximately 1.0×10^{-4} to 3.0 moles/liter. Other details of preparation of water-in-oil-in-water emulsion (i.e. multiple emulsion) can be found in various literatures, such as "Surfactant Systems: Their Chemistry, Pharmacy, & Biology (D. Attwood and A. T. Florence, Chapman & Hall Limited, New York, 1983). If the water-in-oil-in-water emulsion is stable, hollow micro particles with multiple voids will be produced.

The second step (final step) of the said method includes applying heat or radiation to initiate polymerization of the monomers. Useful initiators are free radical polymerization initiators generally used for acrylate or vinyl ester monomers; these initiators are oil soluble, and have a very low solubility in water. However, when the polar monomer is N-vinyl pyrrolidone, benzoylperoxide is suggested as the initiator. The initiators include azo compounds, hydroperoxides, peroxides, and photo-initiators (such as benzophenone, benzoin ethyl ether, and 2,2-dimethoxy-2-phenyl

acetophenone).

Using water soluble polymerization initiator will result in the formation of substantial amount of latex. Extremely small latex particles will result significantly in the formation of undesirable latex. The amount of initiator used is generally in the range of approximately 0.01-10 percent by weight, preferably approximately 5 percent by weight, based on the total weight of the polymerizable composition.

Micro particles may also be prepared by a simpler emulsification process (an "one step process"). The method involves, in the presence of at least one emulsifying agent, polymerization of an aqueous suspension of at least one alkyl methacrylate monomer or vinyl ester monomer and at least one optional polar monomer. The said emulsifying agent is capable of producing water-in-oil emulsion inside the basically stable droplets during the emulsification and polymerization processes. In the said method, the aqueous suspension optionally and preferably also consists of hydrophilic oligomer and/or polymers with free radical reactivity.

In the two step emulsification process, the concentration of the emulsifying agent employed is larger than its critical micelle concentration. Examples of such emulsifying agent include alkyl aryl ester sulfate, such as sodium alkyl aryl ether sulfate, for example, TritonTM W/30 (obtained from Rohm and Haas); alkyl aryl ether sulfates such as alkyl aryl poly-(ethylene oxide) sulfate, preferably those containing up to 4 ethylene oxide repeating units; and alkyl sulfate, such as sodium lauryl sulfate, ammonium lauryl sulfate, triethanolamine lauryl sulfate, and sodium hexadecyl sulfate; alkyl ether sulfates, such as ammonium lauryl ether sulfate, and alkyl polyether sulfates, such as alkyl poly (ethylene oxide) sulfates, preferably those containing up to 4 ethylene oxide units. Alkyl sulfates, alkyl ether sulfates; alkyl aryl ether sulfate and their mixtures are preferred, since they provide a maximum void volume per microsphere with the minimum amount of surface active agent. Non-ionic emulsifiers such as SiponicTM Y-500-70 (ethoxylated oleyl alcohol, obtained from Alcolac, Inc.) and PLURONICTM P103 (a polyethylene oxide and polypropylene oxide block co-polymer, obtained from BASF Company) may be used singularly or in conjunction with anionic emulsifiers. Polymer stabilizer(s) may also be added, however, it is not essential.

Both the "two step" and the "one step" methods produce an aqueous suspension of monomer droplets. The droplets become microparticles during polymerization. Most of the microparticles contain internal cavities. These cavities become voids upon drying.

Another method that may be used to prepare microparticles involves formation of an oil-in-water emulsion; the emulsion is then dispersed in an aqueous phase. The said aqueous phase consists of at least one alkyl (meth)acrylate ester monomer or vinyl ester monomer, and an optional free radical reactive hydrophilic oligomer and/or polymer. The method employs a suspension stabilizer, rather than an emulsifier. As a result, the method is forming micro droplets with no cavity. These micro droplets become solid micro particles upon polymerization.

Segregated polymer micro particles may also be prepared by the suspension polymerization process as described in U.S. Patents 3,691,140; 4,166,152; 4,636,432; 4,656,218; and 5,045,569. The compositions of the adhesive are described in these patents.

The process of preparation of this invention may be improved by withholding the addition of all or portion of the optional free radical reactive hydrophilic oligomer and/or polymer, and optional polar

monomer, prior to initiate the oil phase polymerization. However, before 100 percent conversion, all of the components must be added to the polymerization mixture.

Micro particles may be prepared from many materials. For example , alkyl acrylate and methacrylate monomers may be used to prepare such micro particles. These monomers are mono-functional unsaturated acrylate and methacrylate esters of non-tertiary alcohols. The alkyl groups of these alcohols are preferably having 4-14 carbon atoms. These acrylate ester monomers are oleophilic and water emulsifiable; their water solubilities are quite limited. For homopolymers, their glass transition temperature is generally lower than -10°C. The examples of such monomers include, but not limited to the following groups of monomers: iso-octyl acrylate, 4-methyl-2-pentyl acrylate, 2-methylbutyl acrylate, iso-amyl acrylate, sec-butyl acrylate, n-butyl acrylate, 2-ethylhexyl acrylate, iso-decyl methacrylate, iso-nonyl acrylate, iso-decyl acrylate and their mixtures.

Preferred acrylate ester monomers are selected from iso-octyl acrylate, iso-nonyl acrylate, iso-amyl acrylate, iso-decyl acrylate, 2-ethylhexyl acrylate, n-butyl acrylate, sec-butyl acrylate and their mixtures.

The acrylates, methacrylates, or other vinyl monomers (such as tert-butyl acrylate, iso-borneol acrylate, butyl methacrylate, vinyl acetate, acrylonitrile, and their mixtures) when forming homo polymers with glass transition temperature approximately higher than -10°C, may be optionally used along with one or more acrylate, methacrylate, and vinyl ester, whose glass transition temperature is approximately lower than -10°C when forming polymers.

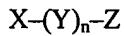
Vinyl ester monomers that are suitable for micro-particles include but not limited to monomers selecting from the following groups: vinyl 2-ethyl hexanoate, vinyl caprate, vinyl laurate, vinyl pelargonate, vinyl hexanoate, vinyl propionate, vinyl caprate (repeated, see above), vinyl octanoate and straight chain or branched carboxylic acid single functional unsaturated vinyl esters containing 1-14 carbon atoms. The glass transition temperatures is approximately lower than -10°C when the said straight chain or branched carboxylic acid single functional unsaturated vinyl esters are forming homopolymers,. The preferred vinyl ester monomers include those selected from the following groups: vinyl laurate, vinyl caprate, vinyl 2-ethyl hexanoate and their mixtures.

The polar monomers used for the preparation of the micro particles are, to certain degree , oil soluble and water soluble; which allow the polar monomers to distribute between the aqueous phase and the oil phase. Typical examples of suitable monomers include but not limited to selection from the following monomer groups: acrylic acid, methacrylic acid, itaconic acid, butanoic acid, maleic acid, fumaric acid, sulfoethyl methacrylate, and ionic monomers such as, sodium methacrylate, ammonium acrylate, sodium acrylate, trimethylamine p-vinyl benzimide, N,N-dimethyl-N-(beta-methoxy-ethyl) ammonium propionate betaine, N-vinyl pyrrolidone, N-vinylcaprolactam, acrylamide, tert-butyl acrylamide, dimethyl-amino-ethyl-acrylamide, N-octyl acrylamide, and their mixtures. Preferred polar monomers include selection from the following monomer groups: mono-olefin monocarboxylic acids, mono-olefin dicarboxylic acids, acrylamides, N-substituted acrylamides, and their salts and mixtures. Examples of such monomers include but limited to acrylic acid, sodium acrylate, N-vinyl pyrrolidone, and their mixtures.

Optional micro particles may also include free radical reactive oligomers and/or polymers. These oligomers and/or polymers include but not limited to poly-alkylene oxides, such as

poly-ethylene oxide, poly-vinyl methyl ether, poly-acrylamide, poly-N-vinyl pyrrolidone, poly-vinyl alcohol, and their mixtures.

The functional derivatives of the free radical reactive hydrophilic oligomers and/or polymers used in the micro particles include macromers with the following general formula:



where: X is a functional group, copolymerizable with free radical polymer monomer and free radical of

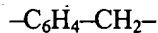
the optional polar monomer;

Y is a divalent linking group;

n is an integer 0 or 1; and

Z is a mono-valent hydrophilic polymer or oligomer with a degree of polymerization equal to or greater than 2.

Examples of such macro monomers include but not limited to: oligomers and/or polymers with acrylate and methacrylate functionality, in which X represents $H_2C=CR^1-$, where R^1 represents H or CH_3 ; Y is a divalent carboxyl group; n=1; and Z is a hydrophilic oligomer or copolymer with a degree of polymerization greater than or equal to 2. Such macromers also include, but not limited to, materials with p-styryl functionality, where X represents $H_2C=CR^1-$, R^1 represents H or CH_3 ; Y represents:



n=1; and Z is a hydrophilic oligomer or copolymer with a degree of polymerization greater than or equal to 2. Di-functional or multi-functional oligomers and/or polymers with more than one X group (such X group acted as a pendent group or a terminal group of the Z segment of the hydrophilic polymers or oligomers) that are capable of free radical polymerization with free radical polymerize monomers and polar monomers of the micro particles of this invention may also be used in the micro particles of this invention.

Preferred macronomers include material selected from the following groups: acrylate terminated poly-ethylene oxide, methacrylate terminated (end capped) poly-ethylene oxide, methoxy-poly-ethylene oxide methacrylate, butoxy-poly-ethylene oxide methacrylate, p-vinyl benzyl terminated poly-ethylene oxide, acrylate terminated poly-ethylene glycol, methacrylate terminated poly-ethylene glycol, methoxy poly-ethylene glycol methacrylate, butoxy poly-ethylene glycol methacrylate, p-vinyl benzyl terminated poly-ethylene glycol, poly-ethylene oxide diacrylate, poly-ethylene oxide dimethacrylate, and their mixtures. Reasons for selecting these functional materials are that they can be easily prepared through well known ionic polymerization techniques, and are also highly efficient in providing grafted hydrophilic chain segment along the main chain of free radical polymerized acrylate polymers.

Preferred macronomers also include material selected from the following groups: p-vinyl benzyl terminated poly-N-vinyl pyrrolidone, p-vinyl benzyl terminated poly-acrylamide, methacrylate

terminated poly-N-vinyl pyrrolidone, and their mixtures; as described in a series of articles by M. Akashi, et al. [Angew. Makromol. Chem., 132, 81 (1958); J. Appl. Polym. Sci., 39, 2027 (1990); J. Polym. Sci., Part A: Polym. Chem., 27, 3521 (1989)]. These maronomers can be prepared through esterification of carboxy terminated N-vinyl pyrrolidone or acrylamide, β -mercapto-propionic acid chain transfer agent and chloromethyl styrene or methylacryloyl chloride; the above articles are referenced in this application.

It is preferable to include at least one polar monomer in the composition, however, the micro particles may also be prepared by using alkyl acrylate, alkyl methacrylate and/or vinyl ester alone or only in combination with other free radical polymerized vinyl monomers (such as vinyl acetate). However, when using alkyl methacrylate alone, a crosslinking agent must be added; unless the hydrophilic component contains more than one free radical co-polymerizable X group as defined in general formula I. It is best (most preferable) to contain at least 1-10 parts by weight of the polar monomers in the composition; such ratio will provide balanced PSA properties for the micro particles.

Compositions for preparing micro particles may also contain multi-functional crosslinking agent. The term "multi-functional" used in this application refers to crosslinking agents that have two or more free radical polymerizable unsaturated vinyl groups. Useful multi-functional crosslinking agents include acrylate or methacrylate esters of diols, such as butanediol diacrylate; triols, such as propanetriol (glycerol); and tetrols, such as pentaerthritol. Other useful crosslinking agents include polymerized esters of multi-functional acrylate and methacrylate, for example, poly-ethylene oxide diacrylate or poly-ethylene oxide dimethacrylate; polyvinyl crosslinking agents, such as substituted or unsubstituted divinyl benzene; and di-functional ethyl urethane acrylates, such as EbecrylTM 270 and EbecrylTM 230 (acrylates of polyurethane with a weight average molecular weight of 1500 and 5000, respectively, obtained from Radcure Specialties) and their mixture(s).

Based on 100 parts by weight, it is preferable that the micro particle used in this invention is consisting of 100 parts or less than 100 parts by weight of at least one free radical polymerizable monomer; the said monomer is selected from alkyl acrylates, alkyl methacrylates, vinyl esters, and their mixtures; optionally, approximately 0-30 parts by weight of one or more polar monomers; and optionally, approximately 0-40 parts by weight of at least one hydrophilic oligomer or polymer component.

Based on 100 parts by weight, it is preferable that the pressure sensitive micro particle used in this invention is consisting of 80-98 parts by weight of free radical polymerizable monomer; the said monomer is selected from alkyl acrylates, alkyl methacrylates, vinyl esters, and their mixtures; optionally, approximately 1-17 parts at least one polar monomers; and optionally, approximately 1-18 parts of hydrophilic oligomer or polymer component. Based on 100 parts by weight, it is preferable that the said pressure sensitive micro particle is consisting of 87-98 parts by weight of free radical polymerizable monomer; optionally, approximately 1-8 parts polar monomers; and optionally, approximately 1-5 parts of hydrophilic oligomer or polymer component.

When using crosslinking agent, the amount employed is often as high as 10 percent equivalent weight. Higher than approximately 0.15 percent equivalent weight (calculated from the total equivalent of the polymerizable micro particles composition) the micro particles become non-tacky. "Percent equivalent weight" of the compound is defined as the number of equivalent weight of the

said compound divided by the total number of equivalent of the entire composition (of the micro particles), in which the number of equivalent is the number (weight) in grams divided by the equivalent weight. The said equivalent weight is defined as the molecular weight divided by the number of polymerizable group in the monomer (when there is only one polymerizable group in the monomer, the equivalent weight is equal to the molecular weight). The crosslinking agent may be added at any time before the said micro particle composition monomers are 100 percent converted to polymers; preferably being added prior to initiation.

The relative amounts of the components in the above composition are important to the properties of the resulting micro particles. Using more multi-functional crosslinking agent will produce non-tacky micro particles. Under these circumstances, water soluble or water dispersible polymer component must be tacky. In the cases where water soluble or water dispersible polymer component itself is not tacky, the adhesive of the micro particles must be tacky.

Water Dispersible Polymer Component

The polymer component used in this invention has sufficient hydrophilic units; these units allow the polymer component to disperse in water. The polymer component preferably contains functional groups that are able to interact with the micro particles. For example, such functional groups include hydroxyl groups, carboxyl groups, amino groups, sulfonyl groups, etc.. It has been found that the adhesive of this invention, using polymer components containing these functional groups, has good shear strength.

Many water dispersible materials may be used as the polymer component of this invention. The non-limiting example of these materials include surface active agents selecting from the following groups: Poly ethylene oxide alkyl phenyl ethers (such as commercially named IGEPAL[®]CO and IGEPAL[®]CA, products obtained from Rhone-Poulenc, Inc.); poly-ethylene oxide lauryl, cetyl, and oleate ethers (such as commercially named Brij[®], products obtained from ICI Americas, Inc.); poly-ethylene oxide laurate, poly-ethylene oxide oleate, sorbitan oleate, and poly-ethylene oxide/poly-propylene oxide block copolymers (such as commercially named PLURONIC[®] and TETRONIC[®], products obtained from BASF Company), and organic phosphate esters (such as Gafac[®] PE-510, product obtained from International Specialty Products).

Other water dispersible polymers that could be used in this invention include polymers described in patents Nos. U.S.2,838,421; U.S.3,441,430; U.S.4,442,258; U.S.3,890,292; U.S.5,196,504 and DE-C23 11 76. Examples of these components include, but not limited to, selection from the following groups of materials: Poly-acrylic acid; poly-vinyl alcohol; poly-N-vinyl pyrrolidone, poly-acrylamide; poly-alkoxyalkyl (meth)acrylates [such as poly-2-ethoxyethyl acrylate, poly-2-ethoxy ethyl methacrylate, poly-2-(2-ethoxyethoxy) ethyl acrylate, and poly-2-methoxy ethyl acrylate (obtained from SARTTOMER Company)]; poly-vinyl methyl ether; poly-(vinyl methyl ether: maleic acid anhydride) (commercially named GantrezTM, products obtained from International Specialty Products); poly-ether polyols, such as poly-propylene-diols (such as commercially named SannixTM, products obtained from Sanyo Chemical Industries); and their co-polymers, etc.. These materials and alkyl (meth)acrylate esters or vinyl ester co-polymers are also suitable. Gums derived from okra and guar tree may also be used.

An especially useful water dispersible polymer contains the pressure sensitive emulsion polymer mentioned above. When an emulsion of the polymer is combined with micro particles, a pressure sensitive composition with excellent shear strength is produced. These emulsion polymers contain polymerization product of poly-alkoxy alkyl acrylate and carboxylic acid. In addition, essentially water insoluble alkyl acrylate and a co-polymerizable emulsion monomer may also be added to the polymer composition; in general, the polymer composition consist of 0-40 parts by weight, preferably 0 part by weight of such alkyl acrylate. The polymer mixture, in general, consists of 0-6 parts by weight, preferably 0-4 parts by weight of the emulsion monomer.

Examples of poly-alkoxy alkyl acrylates used in the preparation of emulsion polymers include, but not limited to: 2-(2-ethoxy ethoxy) ethyl acrylate, 2-ethoxy ethyl acrylate, 2-methoxy ethoxy ethyl acrylate, 2-methoxy ethyl methacrylate, poly-ethylene glycol monoacrylate, and methacrylates, etc..

Examples of carboxylic acids used in preparing emulsion polymers include, but not limited to: acrylic acid, methacrylic acid, itaconic acid, butenoic acid, maleic acid, fumaric acid, β -carboxy-ethyl acrylate, etc..

Example of alkyl acrylates used in the preparation of emulsion polymers include, but not limited to: methyl (meth)acrylate, ethyl (meth)acrylate, n-butyl (meth)acrylate, 2-methylbutyl acrylate, iso-amyl acrylate, sec-butyl acrylate, iso-octyl acrylate,, 2-ethylhexyl acrylate, iso-nonyl acrylate, iso-decyl acrylate, etc..

Example of co-polymerizable surface active agent monomers used in the preparation of emulsion polymers include, but not limited to: anionic vinyl surfactants, such as sodium vinyl sulfonate, sodium styrene sulfonate, alkylene polyalkoxy sulfates, etc..

Another polymer component that may be employed in the micro particles containing adhesive of this invention is described in a pending application No. 08/093,080. The application describes a pressure sensitive adhesive polymer consists of AA, BB, and CC monomers. Where AA monomer is a hydrophobic monomer, with 2-14 carbon atoms, selecting from acrylate or methacrylate esters of non-tertiary alcohol. The AA monomer accounts for approximately 50-80 percent (by weight) of the AA+BB+CC monomers. The BB monomer may be selected from β -carboxy ethyl acrylate (BCEA) or its salts, and mixtures of BCEA and its salts, and other vinyl carboxylic acids or carboxylates. The BB monomer accounts for approximately 10-30 % (by weight) of the AA+BB+CC monomers. If BCEA and vinyl carboxylates or its salts is used, BCEA must account for at least 10 % (by weight) of the polymer. If a mixture of BCEA and vinyl carboxylic acid or its salts is used, BCEA must also account for at least 10 % (by weight) of the polymer. In addition, the carboxylic acid groups in polymer must already been neutralized by an alkali metal hydroxide. The amount of the hydroxide used to neutralize the acid groups is approximately 0.5-2 equivalents of the hydroxide per acid group. The CC monomer is a water dispersible, macronomer with the general formula of X-Y-Z. In the CC monomer, X is the portion that is capable of copolymerizing with AA and BB, Y is a divalent linking group joining X and Z, and Z is a water dispersible group; Z contains at least two elements which essentially will not react under free radical activation co-polymerization conditions (to form polymer). The CC monomer accounts for approximately 10-30 % (by weight) of the AA+BB+CC monomers.

The said pressure sensitive adhesive has an intrinsic viscosity (IV) of approximately 0.2 to greater than 2 dl/g, in 2-butanone at 25°C. Preferably, the intrinsic viscosity is approximately 0.7-1.5

dl/g. Most preferably, the intrinsic viscosity is approximately 0.9-1.4 dl/g.

The AA monomer used in the preparation of terpolymer is an ester of hydrophobic monomer acylate or methacrylate and a non-tertiary alcohol. The said non-tertiary alcohol consists of 2-14 carbon atoms, preferably consists of 4-12 carbon atoms. The non-tertiary alcohol is preferred to be an alkyl alcohol. The term "hydrophobic", used to in this invention, refers to that AA monomer has no significant affinity for water, namely, at room temperature, it essentially neither adsorbs nor absorbs water.

Examples of monomers that are suitable to be used as A monomer include acrylic esters or methacrylic esters of non-tertiary alcohols; such as ethanol, 1-proanol, 2-propanol, 1-butanol, 2-butanol, 1-pentanol, 2-pentanol, 3-pentanol, 2-methyl-1-butanol, 1-hexanol, 2-hexanol, 2-methyl-1-pentanol, 3-methyl-1-pantanol, 2-ethyl-1-butanol, 3,5,5-trimethyl-1-hexanol, 3-heptanol, 1-octanol, 2-octanol, iso-octanol, 2-ethyl-1-hexanol, 1-decanol, 1-dodecanol, 1-tridecanol, and 1-tetradecanol, etc.. Although (a combination of) two or more different AA monomers may also be suitable, but the preferred AA monomers are the butanol or iso-octanol ester of acrylic acid, or their mixtures.

The amount of AA monomer used is preferred to be 50-80 % by weight, based on the total monomer content in preparing the terpolymer. The most preferred amount of the AA monomers used is 60-75 % by weight.

The BB monomers used in the preparation of terpolymer is a polar monomer that is capable of copolymerizing with AA and CC monomers. They are BCEA or its salts, or mixtures of BCEA or its salts and vinyl carboxylic acid such as acrylic acid and its salts. Examples of useful vinyl carboxylic acid include acrylic acid, methacrylic acid, itaconic acid, maleic acid and fumaric acid. BCEA is a commercially available material, it is sold as a mixture of ingredients. For example, BCEA from Rhone Poulenc, Cranbury, NJ is available as a mixture of 20 % by weight acrylic acid, 40 % by weight of BCEA, and 40 % by weight of other acrylic acid oligomers. The amount of BB monomer mixture used in the preparation of polymer is preferably 10-20 % by weight.

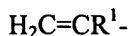
A neutralizer is used in the preparation of such polymer. The amount of neutralizer used is sufficient to neutralize at least 50 % of the acid portion of the copolymer in the adhesive. Excess neutralizer may be used, namely, as high as 2 equivalents of neutralizer per acid group; preferably, 0.75-1.5 equivalents of neutralizer per acid group. Alkali metal hydroxides or mixture of alkali metal hydroxides and a small amount of other neutralizers may be used for such neutralization. It is understandable to the technical personnel who are skilled in the field that many other neutralizing agents may also be used. In order to achieve the desired results, the selection and amount of neutralizer may be varied. However, the selected type and amount used must allow the adhesive to be dispersed. It is preferred to use sodium hydroxide or potassium hydroxide as the neutralizing agent.

The CC monomers used in the preparation of terpolymer are water dispersible macro monomers. Preferably, the CC monomers contain only one vinyl group that is capable of copolymerizing with the AA and BB monomers.

The CC monomers, in general, contains many hydrophilic sites, these sites are giving the

required hydrophilic property to the monomers.

A preferred X group of the CC monomer is a vinyl group with the general formula:



where R¹ is a hydrogen atom or a methyl group.

A preferred Y group of the CC monomer is a divalent carbonyl group.

A preferred Z portion of the CC monomer is a oligomer/polymer material with a molecular weight of 300-30000, preferably 350-5000, most preferably 400-750. The Z portion is preferably to have a poly (ethoxyl oxazoline) or poly (N-vinyl pyrrolidone) or poly alkylene oxide structure. It may be possible to use a Z compound, it is a co-polymer of various compounds such as N-vinyl pyrrolidone and acrylamine. In preparing such co-polymer, only a small amount (less than 10 % by weight) of a monomer, such as acrylamide, is used.

The amount of CC monomer employed is preferably to be 10-30 % by weight of the total weight of the monomers; more preferably, it accounts for 15-25 % by weight.

The ratio of the micro particle component and water dispersible component used in this invention may be varied over a very wide range. For example, the adhesive composition may contain 0.1-99.9 parts by weight of the micro particle composition, and 99.1-0.1 parts by weight of the water dispersible component. It has been found that it is preferable to use a certain narrower range of these two components in special applications. For example, adhesive composition, containing 50-95 (preferably 60-95) parts by weight of micro particle component and 50-5 (preferably 40-5) parts by weight of water dispersible polymer component, is especially useful for preparing splicing tape.

Adhesive compositions contain 10-50 (preferably 10-40) parts by weight of micro-particles and 90-50 (preferably 90-40) parts by weight of water dispersible polymer component are especially useful for sealing systems in surgical dressing, body covers for mammals (such as surgical gowns, sheets, and covers, etc.). For these applications, when such tapes are used, they will not lose their pressure sensitive property during sterilization. A sterilization indicator may also be used at the same time, to indicate that a wrapped package has been through a sterilization cycle. Packages and tapes using the adhesive of this invention are washable, because the adhesive of this invention will disperse in a alkaline laundry solution, therefore, it is not necessary to remove the adhesive before laundering.

Modifying Agents

The adhesive composition of this invention may optionally contain one or more modifying agents, to provide or improve the required properties related to the preparation or application of the adhesive. The amount of modifying agent employed generally is sufficient to produce the desired effect yet is not affecting the pressure sensitive properties of the adhesive. Examples of useful modifying agents include, but are not limited to, thickening agents; such as thickening agent selected from an alkali swellable association polymers; such as commercial products UCAR POLYPHOBE, obtained from Union Carbide, AlcogumTM (obtained from Alco Chemical); RheolateTM (obtained from Rheox); AcrysolTM (obtained from Rhom & Haas); and CarbopolTM (obtained from B. F. Goodrich).

Neutralizers, which are generally used in conjunction with thickening agents or carboxylic acid functional polymers, may also be used in this invention. These neutralizing agents include, but are not limited to metal ions, such as sodium, potassium, calcium, magnesium, copper, aluminum, or zinc; as well as ammonia and organic amines, such as n-hexyl amine, mono ethanol amine, and hexane diamine.

Other modifying agents, that alter the physical properties of the adhesive composition include, but not limited to, tackifying resins, plasticizers, pigments, fillers, flow controlling agents, and stabilizers.

Suitable materials used for flexible carrier support or backing of products of this invention include, but are not limited to, paper, latex saturated paper, polymeric film, cellulose acetate film, ethyl cellulose film, cloth (such as non-woven sheet material from synthetic or natural materials), metal foil, and ceramic thin sheet.

Examples of material suitable for flexible carrier support include polyolefins (such as polyethylene, polypropylene – including isostatic polypropylene), polystyrene, polyester, polyvinyl alcohol, poly ethylene glycol terephthalate, poly butanediol terephthalate, poly hexanolactam, poly (1,2 difluoro ethylene) etc.. Commercially available backing materials used in this invention include kraft paper (brown packaging paper) (obtained from Monadnock Paper, Inc.); cellophane (obtained from Flexel Corp.); spun bond polyethylene and polypropylene, such as TyvekTM and TyparTM (obtained from Dupont, Inc.); and porous films prepared from polyethylene and polypropylene, such as TeslinTM (obtained from PPG Industries, Inc.) and CellguardTM (obtained from Hoechst-Celanese).

Flexible carrier support may also include substrate coated with a release agent. Such substrates are generally used when preparing adhesive transfer tape. Substrate coated with release agent is well known in the field. For example, it includes polysiloxane coated kraft paper, etc..

The tapes of this invention may also include a low adhesion backside (LAB) coating. Such low adhesion backside is generally coated on the backside of pressure sensitive adhesive surface in a tape. Low adhesion backside is well known in the field.

The following application examples are used to illustrate these or those (various) application schemes of this invention. These application examples should not be regard as a limitation to the scope of this invention. The application examples are prepared, and then the following tests are performed, to test their wash-durability, bonding properties, adhesive properties, shear strength and repulping ability.

Test Methods

Bonding Properties

In the said test, a Delrin wheel (weighing 29 grams), with a diameter of 8.13 cm, and a wheel width of 1.9 cm, is rolled down along a surface 26.7 cm long, at a horizontal incline angle of 24°; a test tape is placed on the said horizontal surface with the adhesive side facing up. The distance of the wheel traveled along the horizontal surface is measured. The bonding properties are inversely proportional to the rolling distance of the wheel.

Repulping Ability

The repulping ability is measured according to TAPPI test UM-213. For a double sided tape, a 20 cm x 2.54 cm tape strip is sandwiched between two 20 cm x 2.54 strips of blotter paper. For a single sided tape, two 20 cm x 2.54 tape strips are attached to a blotter paper. The samples are cut into approximately 1.5 cm² squares. A sufficient number of 1.5 cm² blotter paper pieces are then added to the tape/blotter paper bonded pieces, to reach a total test sample weight of 15 g. The samples are then placed in a "Weilin" blender along with 500 ml of room temperature tap water. After the blender has run for 20 seconds, it is stopped for 1 minute, wash the paper material that is splashed onto the side and cover back to the bottom with a water bottle. The blender is then run for 20 more seconds, and again washes the splashed paper back down by the method mentioned above; the blender is then run for final 20 seconds, and the paper stock is removed from the blender, a paper towel is then prepared in a paper making mold. The paper towel is removed from the mold, and placed between blotting paper for 90 seconds with a hydraulic press; it is then dried and inspected for any particles of un-repulped tape. If there are only two or a few particles, it is considered that the tape is passing the test. The presence of one or two particles is not considering a failure because they may be coming from contaminated equipment or screens. When a material is failing the test, particles will be dispersed over the entire towel.

The above test is not suitable to apply to tape products that are using a non-repulpable backing such as polyester film. The dispersion capability of the adhesive on these baking may be measured by using the methods for measuring adhesive transfer tapes or an adhesive film on a water dispersible carrier layer.

Laundering Test

A piece of tape to be tested is laminated to an untreated 50 % cotton/50 % polyester fabric (obtained from Standard Textile Co., Cincinnati, OH, commercially referred to as Barrier SupremeTM), to form a test sample. A 1.8 kg (4 lbs) rubber roller is used to laminate the tape to the said fabric, allowing the adhesive containing micro particles to be exposed. The test sample is then steam sterilized at 134°C, using a 10 minute 4 pulse pre-vacuum cycle and 1 minute steam drying.

The test sample is then placed in a 60 lb commercially available washing machine (Milnor washer, Model No. 36021 BWE/AEA; Pillerin Milnor Corp., Kenner, LA) and washed, allowing the sample to go through a general surgical linen washing cycle, the said cycle include: (a) cold alkaline wash for 3 minutes using 0.1 % Paralate 55 GL11TM (Ecolab Inc., St. Paul, MN) commercial liquid laundry soda; 3 minutes of cold water rinse, and 8 minutes of hot break/suds wash using 0.1 % Paralate 55 GL11TM (Ecolab Inc., St. Paul, MN) commercial liquid laundry soda and 0.05 % KindetTM (Ecolab Inc.) commercial laundry liquid; two 3-minute hot water rinsing; one 3-minute cold water rinsing; 3 minutes of cold sour/soft rinse using 0.05 % Tri Liquid Sour 55 GLTM commercial liquid laundry sour (Ecolab Inc.) and 0.05 % Tex Special LiquidTM commercial liquid cloth lubricant (Ecolab Inc.); and 6 minutes of pressing and squeezing to remove excess liquid.

Each test sample is inspected; fabric with no adhesive residue is passing the test, while those with adhesive residue are failing the test.

Adhesion

This test is measuring an 180° peel adhesion against paper. A 3.175 cm bond paper is laminated to a 5 cm x 12.5 cm stainless steel test panel using a double coated tape. One end of a 2.54 cm x 25 cm tape test sample is attached to the bond paper (if the test sample is double sided tape, one side of the adhesive of the test sample is cover with 68 g/m² kraft paper). A 2 kg roller is used to roll over the 12.5 cm long test sample at a speed of 30.6 mm/min, to assure uniform contact of the adhesive. The test sample is then folded against itself, and peeled off at a speed of 30.5 cm/min from the bond paper. An Instron, Model 1122, operating at 21°C under 50 % relative humidity, is used to record the force in peeling off the tape.

Shear Strength

Use 2.54 cm x 2.54 cm end portion of a 2.54 cm x 15 cm tape; if the said tape has adhesive only on one side, the adhesive is adhered to a bright annealed steel test panel, and use a 2 kg roller to roll over twice. If the tape has adhesive on each surface, the adhesive, whose shear strength is being tested, is adhered to a bright annealed steel test plate, a piece of 68 g/m² super smooth and clean finished kraft paper backing is then laminated to the other adhesive surface. The steel plate is clamped to a fixture that is 2° from vertical, allowing the free end of the tape to extend downward at an angle of 178° from the test panel; a 1000 g weight is attached to that end. Time (to the nearest minute) the tape separated from the test panel is recorded, shear strength is directly correlated to the time elapsed. Refer to test method, Test PSTC-7, for pressure sensitive tape mentioned above for other details. For tapes prepared from the adhesive of this invention, it is required to have as long a shear time as possible, preferably more than 1000 minutes; although, an adhesive may still be satisfactory even with a shear time as low as 40 minutes.

Abbreviations

The following abbreviations are used in the Application Examples described in this application.

AA = acrylic acid

BA = butyl acrylate

BCEA = β-carboxyethyl acrylate

PEOA = poly (ethylene oxide) monoacrylate, average molecular weight 750

EOEA = 2-ethoxy ethyl acrylate

EOEOEA = 2-(2-ethoxy) ethoxy ethyl acrylate

HDDH (A ?, also see below Micro Particles C, D preparations) = 1,6-hexanediol diacrylate

IOA = isoctyl acrylate

MSA = micro particle adhesive

PC = water dispersible polymer component

PPG = poly propylene glycol

PAA = poly acrylic acid

PVA = poly vinyl alcohol

Repulp = repulpability test

P = pass (repulpability test)

F = fail (repulpability test)

The expressions inside the parenthesis at the beginning of each preparation indicate the monomer components and the weight percent of each component used in the preparation.

Preparation of Micro Particles

Micro Particles A (IOA:AA:PEOA/97:2:1)

4.8 g of AA, 2.4 g of PEOA, and 1.13 g of LucidalTM-70 (70 % benzoyl peroxide, obtained from Elf Atochem) are dissolved in 232 g of IOA. 0.75 g of SiponateTM DS-10 (sodium dodecyl benzene sulfonate surfactant, obtained from Phone-Poulenc) is dissolved in 360 g of water. The IOA mixture is then added to the surfactant solution, and emulsified with an OmniTM mixer, until droplet size is smaller than 5 microns. The emulsion with 40 % solid is poured into a 1 liter baffled reactor, heated to 65°C, purged with nitrogen, and allowed to react for 8 hours.

Micro Particles B (IOA:AA:PEOA/89:1:10)

2.1 g of AA, 21 g of PEOA, and 0.21 g of poly (ethylene oxide)₉ dimetharylate, and 0.99 g of LucidolTM-70 are dissolved in 189.5 g of IOA; 6 g of StandapolTM (a surfactant, namely, ammonium lauryl sulfate, Hercules) is dissolved in 390 g of water. The IOA mixture is then added to the surfactant solution, and emulsified with an OmniTM mixer, until droplet size is smaller than 5 microns. The emulsion is poured into a 1 liter indented resin flask, heated to 65°C, purged with argon, and allowed to react for 18 hours.

Micro Particles C (IOA:BA:AA:PEOA/79:17:1:3)

2.1 g of AA, 8.4 g of PEOA, 39.9 g of BA, 0.25 g of HDDA, and 0.99 g of LucidalTM-70 are dissolved in 189.9 g of IOA; 6.5 g of StandapolTM (a surfactant, namely, ammonium lauryl sulfate, Hercules) is dissolved in 390 g of water. The IOA mixture is then added to the surfactant solution, and emulsified with an OmniTM mixer, until droplet size is smaller than 5 microns. The emulsion is poured into a 1 liter indented resin flask, heated to 65°C, purged with argon, and allowed to react for 22 hours.

Micro Particles D (IOA: AA:HDDA/96:2:2)

4.2 g of AA, 4.2 g of HDDA, and 1.13 g of LucidalTM-70 are dissolved in 230 g of IOA; 0.75 g of ammonium lauryl sulfate surfactant is dissolved in 360 g of water. The IOA mixture is then added to the surfactant solution, and emulsified with an OmniTM mixer, until droplet size is smaller than 5 microns. The emulsion is poured into a 1 liter indented resin flask, heated to 65°C, purged with argon, and allowed to react for 12 hours; the micro particles obtained are not tacky.

Micro Particles E (100 % IOA)

The micro particles are prepared in a 1 liter indented resin flask containing 450 ml of deionized water and 4 g of StandapolTM. The said solution is stirred at 400 rpm, heated to 70°C, purged with argon, then 150 g of IOA and 0.71 g of LucidalTM-70 are added to the hot surfactant solution. The temperature is decreased to 65°C, and allowed to react for 22 hours. Measuring with an optical

microscope, the average particle size is approximate 33 microns.

Preparation of Water Dispersible Polymer Components

PC-1 (EOEOEA:AA/80:20)

1217 g of deionized water and 0.39 g of potassium persulfate are added to a 3 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 150 rpm.. Preparation charges I, II, and III are prepared; charge I consists of 13.5 g of Mazon™ SAM 211 (obtained from PPG/Mazer Chemicals) dissolved in 100 g of deionized water; charge II consists of 0.39 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 240 g of EOEOEA, 60 g of AA and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-2.5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 35 minutes; the emulsified content is cooled, and poured from the flask. The pH is 3.32, Brookfield viscosity = 5 cps, and the particle size is 182 μ .

PC-2 (EOEA:AA/85:15)

800 g of deionized water and 0.39 g of potassium persulfate are added to a 2 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 300 rpm. Preparation charges I, II, and III are prepared; charge I consists of 13.5 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; charge II consists of 0.39 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 255 g of EOEOEA, 45 g (of AA ?) and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-2.5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 35 minutes; the emulsified content is cooled, and poured from the flask. The particle size of the polymer is 94.3 μ .

PC-3 (EOEOEA:BA:AA/75:15:10)

800 g of deionized water and 0.39 g of potassium persulfate are added to a 2 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 300 rpm.. Preparation charges I, II, and III are prepared; charge I consists of 13.5 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; charge II consists of 0.39 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 255 g of EOEOEA, 45 g of BA, 30 g of AA, and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-2.5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 35 minutes; the emulsified content is cooled, and poured from the flask. The particle size of the polymer is 236 μ .

PC-4 (EOEOEA:BA:AA/70:15:15)

1672 g of deionized water, 20.9 g of Mazon™ SAM-211 and 2.1 g of potassium persulfate are added to a 3 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 200 rpm.. Prepare a pre-mixture consists of 292.6 g of EOEOEA, 62.7 g of BA, 62.7 g of AA and 0.30 g of tert-dodecyl mercaptan. The mixture is added to the flask using a dropping funnel over 5 hours. The emulsified content is allowed to cool, and poured from the flask. The pH = 2.46, Brookfield viscosity = 4.5 cps, and the particle size is 108 µ.

PC-5 (EOEOEA:BA:AA/50:35:15)

1672 g of deionized water, 20.9 g of Mazon™ SAM-211 and 2.1 g of potassium persulfate are added to a 3 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 200 rpm.. Prepare a pre-mixture consists of 209 g of EOEOEA, 146.3 g of BA, 62.7 g of AA and 0.21 g of tert-dodecyl mercaptan. The mixture is added to the flask using a dropping funnel over 6 hours. The emulsified content is allowed to cool, and poured from the flask. The pH = 2.55, Brookfield viscosity = 4.5 cps, and the particle size is 91.0 µ.

PC-6 (EOEOEA:BCEA/82:18)

2000 g of deionized water and 0.65 g of potassium persulfate are added to a 3 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 150 rpm.. Preparation charges I, II, and III are prepared; charge I consists of 22.5 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; charge II consists of 0.65 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 410 g of EOEOEA, 90 g of BCEA and 0.15 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 35 minutes; the emulsified content is cooled and poured from the flask. The pH = 4.47, Brookfield viscosity = 1.5 cps, and the particle size is 146 µ.

PC-7 (EOEOEA:BA:AA/60:20:20)

1217 g of deionized water and 0.39 g of potassium persulfate are added to a 3 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 150 rpm.. Preparation charges I, II, and III are prepared; charge I consists of 13.5 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; charge II consists of 0.39 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 180 g of EOEOEA, 60 g of BA and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-2.5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 35 minutes; the emulsified content is cooled, and poured from the flask. The particle size of the emulsified reaction product is 101 µ, and the Brookfield viscosity = 5 cps.

PC-8 (EOEOEA:IOA: β -CEA/50:17:33)

1200 g of deionized water and 0.39 g of potassium persulfate are added to a 3 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 150 rpm.. Preparation charges I, II, and III are prepared; charge I consists of 13.5 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; charge II consists of 0.39 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 150 g of EOEOEA, 51.0 g of IOA, 99.0 g of BECE, and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-3 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 35 minutes; the emulsified content is cooled, and poured from the flask. The pH value of the emulsified reaction product = 3.32, and the Brookfield viscosity = 3 cps, and the particle size is 201 μ .

PC-9 (EOEOEA: AA/95:5)

800 g of deionized water and 0.39 g of potassium persulfate are added to a 2 liter split resin flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, and stirred at a speed of 150 rpm.. The flask is purged with nitrogen at a rate of 1 liter per minute. Preparation charges I, II, and III are prepared; charge I consists of 15.0 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; charge II consists of 0.39 g of potassium persulfate dissolved in 50 g of deionized water; and charge III consists of 285 g of EOEOEA (obtained from Sartomer Chemical Co.), 15 g of AA, and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II is then added gradually over 1-2.5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 30 minutes; the emulsified content is cooled, and poured from the flask.

PC-10 (IOA:AA/85:15)

800 g of deionized water and 0.39 g of potassium persulfate are added to a 2 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen (at 1 l/min), and stirred at a speed of 150 rpm.. Preparation charges I and II are prepared; charge I consists of 13.5 g of Mazon™ SAM 211 dissolved in 100 g of deionized water; and charge II consists of 225 g of IOA, 45 g of AA, and 0.09 g of tert-dodecyl mercaptan. Charge I and 75.0 g of charge II are added to the flask, reacted for 30 minutes at 76°C. The remainder of charge II is then added over 2 hour. The content in the flask is allowed to react for 30 minutes; the emulsified content is cooled, and poured from the flask. The emulsified reaction product has a solid content of 26.8 % by weight, and the particle size is 128 nm (μ).

PC-11 (EOEOEA:AA/90:10)

2403 g of deionized water and 1.17 g of potassium persulfate are added to a 5 liter flask, equipped with condenser, stirrer, and nitrogen purge line. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 150 rpm.. Preparation charges I, II, and III are prepared; charge I consists of 40.5 g of Mazon™ SAM 211 dissolved in 297 g of deionized water; charge II consists of

1.17 g of potassium persulfate dissolved in 100 g of deionized water; and charge III consists of 810 g of EOEOEA, 90 g of AA and 0.27 g of tert-dodecyl mercaptan. Charge I and 300 g of charge III are added to the flask, reacted for 30 minutes. The remainder of charge III and all of the charge II are then added gradually over 1-2.5 hour to the flask. After adding all the charges, the content in the flask is allowed to react for 30 minutes; the emulsified content is cooled, and poured from the flask.

PC-12 (EOEOEA:AA/90:10)

62.3 kg of deionized water and 21.2 g of potassium persulfate are added to a 25 gallon (95 liter) porcelain enamel (glass lined) reactor. The mixture is heated to 76°C, purged with nitrogen, and stirred at a speed of 75 rpm.. Preparation charges I and II are prepared; charge I consists of 1.6 kg of AA, 735 g of Mazon™ SAM 211, 14.7 kg of EOEOEA and 4.9 g of tert-dodecyl mercaptan; charge II consists of 5.3 kg of deionized water and 21.2 g of potassium persulfate. 4.3 kg of charge I is added to the flask (? reactor), reacted for 30 minutes. The remainder of charge I and all of the charge II are then added gradually over 1-2.5 hour. After adding all the charges, the content in the flask (? reactor) is allowed to react for 1 hour. The emulsified reaction product is cooled, and poured (? discharged) from the flask (? reactor). The emulsified reaction product has a particle size is 228 μ , and the Brookfield viscosity = 4 cps.

Application Examples 1 – 10

Adhesive test samples are prepared through mixing of micro particles with 1 % by weight of an associative alkali swellable thickener (UCAR Polyphobe™ 104, obtained from Union Carbide) using only micro particle components A-E, which are then neutralize with potassium hydroxide and poly (oxypropylene) tert-amine, to a pH of 7. The adhesive is coated on a release surface, then dried in a 79°C oven for 3 minutes; laminated to a “Crystex” thin paper, to obtain a single sided structure. To obtain a double sided structure, adhesive is coated on the opposite side of the single sided test sample, and dried in the similar manner. Test is conducted after the release lining is removed.

Adhesive tape test samples, for Application Examples 1–10, are prepared as follows: Mix together micro particle component A with a necessary water soluble or water dispersible polymer component, then use the similar methods to thicken, neutralize, and coat as the method mentioned above in preparing tapes with micro particle components A-E. The water soluble or water dispersible polymer components are organic phosphate Gafac® PE-510 obtained from International Specialty Products; polyvinyl alcohol, with a molecular weight 8×10^5 g/mol; poly acrylic acid PAA, with a molecular weight of 2×10^5 g/mol; polyethylene oxide PEO, with a molecular weight of 10^6 g/mol; ethoxyalkylphenols Igepal® CA-520, CA-630, and C-630 (CO-630?, see Table 1 below) , obtained from International Specialty Products; polyalkylene glycol Sannix® SP-750, obtained from Sanyo Chemical Industries, and polypropylene glycol PPG, with a molecular weight of 400 g/mol.

These application examples demonstrate that: addition of water soluble or water dispersible polymer component to the micro particle adhesive increases its tackiness while maintaining its repulping ability. These results are listed in Table 1.

Table 1

Application Example	Polymer Component	Percent P.C.	Tackiness (mm)	Repulping Test (P/F)
Comparison	None (all micro particle component A)	0	80	P
1	Gafac® PE-510	0.5	15	P
2	Gafac® PE-510	1.0	20	P
3	PVA	1.0	33	P
4	PVA	1.0	38	P
5	PEO	1.0	29	P
6	Igepal® CA-520	2.5	48	P
7	Igepal® CA-630	2.5	47	P
8	Igepal® CO-630	2.5	36	P
9	Sannix® SP-750	5.0	30	P
10	PPG	5.0	33	P

Application Examples 11-21

Application Examples 11-21 are prepared, with the similar methods as described in Application Examples 1-10, using micro particles component A and polymer components listed in Table 2, but without adding a thickening agent.

These application examples demonstrate that: the adhesion performance is improved in all cases when the water soluble or water dispersible polymer component is a PSA, because in the majority of cases, it is tacky, while the repulping ability is being maintained. The results are listed in Table 2.

Table 2

Application Example	Polymer Component	% PC Component	Tackiness (mm)	Repulping Test (P/F)	Adhesion (N/cm)
Comparison	None (all micro particle A)	0	80	P	3.6
11	PC-1	22	56	P	6.9
12	PC-2	22	17	P	8.0
13	PC-3	22	20	P	7.3
14	PC-4	22	123	P	7.1
15	PC-5	22	79	P	6.9
16	PC-6	22	14	P	8.4
17	PC-7	13	38	P	6.3
18	PC-8	13	20	P	7.1
19	PC-1	13	33	P	6.4
20	PC-9	13	21	P	5.8
21	PC-10	22	32	P	6.0

Application Examples 22-23

In Application Examples 22-23, PC-11 is used as the water soluble or dispersible polymer component, the microparticle is microparticle component A. Application Example 22 is prepared using similar method as described in Application Example 1. Application Examples 23-32 is prepared using similar method as described in Application Example 11. Application Example 33 is neutralized using similar method as described in Application Example 1.

These application examples demonstrate the properties of this invention over a wide range of polymer component concentration range. As shown in Table 3, the results indicate that, comparing to Comparison Example A, the tackiness, adhesive and shear performance are improved (in most cases); at the same time, the repulping ability is maintained.

Table 3

Application <u>Example</u>	Polymer <u>Component %</u>	Tackiness (mm)	Repulping Test (P/F)	Adhesion (N/cm)	R.T. Shear Adhesion (min)
Comparison	0 (all micro particle component A)	80	P	3.6	1330
Example A					
22	5	62	P	5.4	2800+
23	92	37	P	5.4	2800+
24	13	24	P	5.4	2800+
25	23	21	P	6.1	2800+
26	31	18	P	6.1	2800+
27	38	18	P	5.7	2800+
28	43	22	P	6.1	2020*
29	47	22	P	6.5	1650*
30	60	35	P	5.7	130*
31	75	33	P	5.9	76*
32	90	39	P	5.9	57*
33	100 (all PC-11)	31	P	7.8	130*

* indicates shear

Comparison Example A suddenly falling off at 1330 minutes. Application Examples 28, 29, and 33 sheared at the time indicated.

Application Examples 34-38

These application examples demonstrate the effect of different ratio of EOEOEA and acrylic acid copolymer water dispersible polymer component with different types of micro particles adhesive mixture. Application Examples 34, 35, 36, 37, and 38 (containing polymer component) are prepared in the same manner as described in Application Example 11.

Comparison Example B and Application Example 34 are comparing micro particles composition containing 10 % of PEOA. Comparison Example B uses no polymer component. Application Example 34 uses 22 % by weight of PC-5. As shown in Table 4, addition of PC-5 improves adhesion and repulping ability.

Table 4

<u>Application Example</u>	<u>Polymer Component</u>	<u>PC Component</u>	<u>Tackiness (mm)</u>	<u>Repulping Test (P/F)</u>	<u>Adhesion (N/cm)</u>
Comp. Examp. B	B	...	19	F	5.8
34	B	PC-5	35	P	6.7

Comparison Example C and Application Example 35 are comparing micro particles composition containing PEOA and butyl acrylate. Comparison Example C uses no polymer component. Application Example 35 uses 22 % by weight of PC-1. As shown in Table 5, addition of PC-1 improves adhesion and repulping ability.

Table 5

<u>Application Example</u>	<u>Polymer Component</u>	<u>PC Component</u>	<u>Tackiness (mm)</u>	<u>Repulping Test (P/F)</u>	<u>Adhesion (N/cm)</u>
Comp. Examp. C	C	...	33	F	3.7
35	C	PC-1	180	P	5.6

Comparison Example D and Application Examples 36 and 37 are comparing micro particles composition that is not tacky. Comparison Example D uses no polymer component. Application Examples 36 and 37 use 43 % by weight of PC-4. As shown in Table 6, addition of PC-4 improves adhesion and repulping ability.

Table 6

<u>Application Example</u>	<u>Polymer Component</u>	<u>PC Component</u>	<u>Tackiness (mm)</u>	<u>Repulping Test (P/F)</u>	<u>Adhesion (N/cm)</u>
Comp. Examp. D	D*	P	...*
36	D	PC-4	150	P	1.3
37	D	PC-4	150	P	2.9

* Not tacky, can not be tested.

Comparing to other application examples (diameters, approximately 3 microns) described in this application, Comparison Example E and Application Example 38 use a relatively larger particle size (diameter, 33 microns) micro particle composition. Comparison Example uses no polymer component. Application Examples 38 uses 43 % by weight of PC-12. Addition of PC-12 improves adhesion and repulping ability.

Table 7

<u>Application Example</u>	<u>Polymer Component</u>	<u>PC Component</u>	<u>Tackiness (mm)</u>	<u>Repulping Test (P/F)</u>	<u>Adhesion (N/cm)</u>
Comp. Examp. E	E	...	170*	F	6.1
38	E	PC-12	130	P	4.1

Application Examples 39-42

The pressure sensitive adhesive tapes of this invention are prepared, laminated to an untreated 50 % cotton/50 % polyester fabric (Barrier Supreme™), and then washed as the described in the Laundering Test Method. The backing of the tape is "Crystex" thin paper. The adhesive compositions and test results are given in Table 8.

Table 8

<u>Application Example</u>	<u>Micro Particle Component</u>		<u>Water Dispersible Component</u>	
	<u>Material</u>	<u>Parts by Weight</u>	<u>Material</u>	<u>Parts by Weight</u>
39	A	78	PC-6	22
40	A	87	PC-7	13
41	A	87	PC-1	13
42	A	87	PC-8	13

There is no evidence of residue on the washed fabric in all of the test samples.

Application Example 43

A water dispersible polymer is prepared. A mono-methoxy polyethylene glycol monomer, with a molecular weight of 750, is prepared through Fischer esterification of Carbowax™ 750 (Union Carbide). 100 parts of Carbowax™ 750 and 100 parts of methyl benzene is placed in a flask equipped with a Dean-Stark condenser and a stirrer. The said solution is heated at reflux temperature for 2 hours. 11.3 parts of acrylic acid, 4.5 parts of toluene-p-sulfonic acid, 3000 ppm of Irganox™ PS 800 (Ciba-Geigy), and 500 ppm of thiadiphenylamine are added to the mixture. The solution is again heated at reflux temperature for 16 hours. Cool to room temperature, and the excess acid is neutralized with 4.5 parts of calcium hydroxide. The precipitate formed is filtered off. The methyl

benzene is then evaporated under reduced pressure to obtain a 100 % solid acrylate monomer.

A water dispersible pressure sensitive adhesive polymer is prepared as follows: Butyl acrylate, BCEA (a mixture of 20 % by weight acrylic acid, 40 % BCEA, and 40 % by weight of other acrylic acid oligomers available commercially from Rhone Poulenc), and mono-methoxy polyethylene glycol acrylate monomer are added to a 1 quart bottle containing a solvent mixture of ethyl acetate, methanol, iso-propanol and 0.51 g of azodiisobutyronitrile; the monomer input material contains 32-34 % polymer mixture. It is purged with nitrogen at a rate of 1 liter/minute for 2 minutes, to deoxygenate the mixture. The bottle is sealed and placed in a 55°C rotating water bath for 24 hours, allowing it to essentially polymerize completely. The intrinsic viscosity (IV) of the resulting polymer is measured in 2-butanone at 27.5°C. Neutralization agent (86.8 % pure KOH, calculated based on 100 % pure KOH, 1.25 neutralization equivalents) in 3.75 N 50 : 50 methanol : water (V/V) solution, is then added to the said adhesive polymer. Micro particles (A) are then added to the adhesive polymer, obtaining a final ratio of 20 g of micro particles to 100 g adhesive polymer (namely, 16.7 % by weight of micro particles).

The resulting neutralized pressure sensitive adhesive composition containing micro particles is then coated on a 29 lb base weight kraft paper (M-2383 Smooth Crepe Semi-Bleached Kraft Saturating Paper, manufactured by Mosinee Paper Corporation of Mosinee, Wisconsin), forming the following sealing tape.

Using a standard laboratory knife coater, the pressure sensitive adhesive, dissolved in a solvent system, is coated on the kraft paper at 0.68 g/154.8 cm² (24 in²), then dry in a 100°C forced (air) oven for 15 minutes.

The initial adhesion, adhesion after sterilization, package sealing before sterilization – T(0) , package sealing after sterilization – T(A), and dispersion ability of Application Example 43 and Comparison Example 1, with coated fluoro-chemical treated polyester packaging paper, are given in Table 9.

Initial Adhesion of treated Surgical Drape Wrapper

Fluoro-chemical treated polyester surgical drape (obtained from Standard Textile Co., Cincinnati, OH as WrapPel™ T) test samples are cut into 3.81 cm x 10.16 cm tapes, and # 410 double coated tape (obtained from 3M Company) is used to attach to a 5.08 cm x 12.2 cm steel plate. The application example sealing tapes (2.54 cm x 10.16 cm) of this invention are adhered to the said drape, and rolled over twice with a 2 kg rubber roller.

Place one end of each steel plate into a set of clamps of an Instron Model 1122 Tensile Tester (Instron Corp., Canton, MA), the other end of the sealing tape is adhered to another set of clamps of the tensile tester, allowing the tape to be peeled off from the drape essentially at an 180° angle. The speed that the clamps are moving is 30.48 cm/min, the tensile tester record the force required to peel off the tape laminated to the drape. The results are recorded in Newtons/2.54 cm (N/2.54 cm). The initial adhesion measurement is based on the fact that the tape has very little dwell time on the drape, namely the attachment of application example test tape and its peeling off has very little residence time (approximately less than 5 minutes), and sterilization has not yet taking place.

Adhesion of Treated Tape after Sterilization

Except prior to testing, the application example sealing test tape is steam sterilized at 273°F (134°C) for 10 minutes using a gravity cycle, then steam dry for 3 minutes, the method used in this test is similar to that of the initial adhesion test. The sterilizer used is Barnstead Model GLS-10D (MDT Biologic Co., Rancho Dominguez, CA). In addition, the laminate is allowed to cool at room temperature for at least 4 hours. The results are recorded as N/2.54 cm.

Static Shear Strength

The static shear strength of the tape is tested using the following method. A stainless steel plate is washed with diacetone, wiped with tissue paper (KIM-WIPE), after washing in heptane and wiping with tissue paper, it is again rinsed twice in heptane. A section of tape is attached to a WrapPel™ drape material, obtaining a 2.54 cm x 2.54 cm bonding area. The other portion of the tape is hanging over the edge, and is folding back into a ring. Allowed a 2 kg roller to roll back and forth once over the bonded area along the length of the tape. A 250 g weight is tie to the ring, and allowed the device to hang down vertically at a room maintaining at 20°C and 50 % relative humidity. The failure (breaking) time is measured (the time falling from the stainless steel plate).

Table 8

Property

Initial Adhesion	229 N/2.54 cm
Adhesion after Sterilization	88N/2.54 cm
Static Shear Strength	88 minutes
Dispersion Ability	Pass

Although the invention has been described through specific application examples, it should be understood that further modification is possible for this invention. The claims are attempting to cover chemical equivalent of all variations of this invention that can be recognized by the technical personnel who are skilled in the field.

[19]中华人民共和国专利局

[51]Int.Cl⁶

C09J133/08

C09J 7/02



[12]发明专利申请公开说明书

[21]申请号 95192420.6

[43]公开日 1997年3月12日

[11]公开号 CN 1145087A

[22]申请日 95.2.24

[74]专利代理机构 上海专利商标事务所

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[32]94.4.4 [33]US[31]08 / 222,458

[86]国际申请 PCT / US95 / 02295 95.2.24

[87]国际公布 WO95 / 27016 英 95.10.12

[85]进入国家阶段日期 96.10.3

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权利要求书 3 页 说明书 24 页 附图页数 0 页

[54]发明名称 压敏粘合剂及其制品

[57]摘要

本发明提供一种使用水分散性聚合物的压敏粘合剂。在一个实施方案中该粘合剂含有微粒和水分散性组分。在另一个实施方案中，该粘合剂含有以大量聚丙烯酸烷基烷酯为主要组分的新颖水分散性聚合物。该聚合物可用作水分散性组分。压敏粘合剂可制成带和用作容器的标签、消毒指示带和标签、信封的密封系统、外科包扎带和哺乳动物身体覆盖物。它也可用于制造纸卷材拼接物、在一个实施方案中粘合带是可再浆化的，在另一个实施方案中它是可洗涤的。

(BJ)第 1456 号

权 利 要 求 书

1. 一种通常粘着性的压敏粘合剂组合物，它含有如下组分的混合物：
 - A) 不溶于溶剂但可被溶剂分散的弹体聚合物微粒组分；
 - B) 水分散性聚合物组分。
- 5 2. 如权利要求 1 所述的压敏粘合剂组合物，其特征在于含有 0.1-99.9 重量份所述的微粒组分，和相应地 99.9-0.1 重量份所述的水分散性组分。
3. 如权利要求 1 所述的压敏粘合剂组合物，其特征在于所述微粒组分的微粒部分或者是实心颗粒，或者是至少含一个空隙的颗粒。
- 10 4. 如权利要求 3 所述的压敏粘合剂组合物，其特征在于：
 - (A) 微粒组分是含如下组分的聚合物：
 - (1) 100 或小于 100 重量份的至少一种可自由基聚合的单体，该单体选自丙烯酸烷酯、甲基丙烯酸烷酯、乙烯基酯及其混合物；
 - (2) 0-30 重量份的至少一种可与自由基聚合单体 A(1)共聚的极性单体；
 - (3) 0-40 重量份至少一种可与自由基聚合单体 A(1)或极性单体 A(2)共聚的亲水性低聚物或聚合物组分；
 - (B) 水分散性聚合物组分是含如下组分的聚合物：
 - (1) 50-98 重量份水溶性聚丙烯酸烷氧基烷酯，以及
 - (2) 50-2 重量份羧酸。
- 20 5. 如权利要求 4 所述的压敏粘合剂组合物，其特征在于微粒组分含 80-98 份可自由基聚合单体、1-17 重量份极性单体和 1-18 重量份亲水低聚物或聚合物。
6. 如权利要求 5 所述的压敏粘合剂组合物，其特征在于所述亲水组分含通式如下的大单体：

25 $X-(Y)_n-Z$

式中

X 是与可自由基聚合单体和任选的极性单体自由基共聚的基团；

Y 是两价连接基团；

30 n 是 0-1 的整数；

Z 是聚合度大于或等于 2 的单价亲水性聚合物或低聚物部分。

7. 如权利要求 5 所述的压敏粘合剂组合物，其特征在于水分散性聚合物组分还含有 0-40 重量份基本上非水溶性的丙烯酸烷基酯和 0-6 重量份乳化剂单体。

8. 如权利要求 7 所述的压敏粘合剂组合物，其特征在于 80-95 重量份水溶性聚丙烯酸烷氧基烷酯、20-5 重量份所述羧酸和 0-4 重量份所述乳化剂单体。

35 9. 一种压敏粘合剂片材，它包括一层如权利要求 1 所述的通常粘着性的压敏

粘合剂.

10. 如权利要求 9 所述的压敏粘合带, 其特征在于所述通常粘着性的压敏粘合剂片材涂在柔性载体的至少一个表面.

11. 如权利要求 10 所述的压敏粘合带, 其特征在于所述通常粘着性的压敏粘合剂层可剥离地粘接于所述的柔性载体.

12. 如权利要求 10 所述的压敏粘合带, 其特征在于所述通常粘着性的压敏粘合剂层基本上永久地粘接于所述的柔性载体.

13. 如权利要求 12 所述的压敏粘合带, 其特征在于所述的带是可再浆化的.

14. 一种可再浆化的压敏粘合带, 含有一薄层如权利要求 1 所述的通常粘着性的压敏粘合剂组合物, 其特征在于微粒组分是悬浮聚合材料.

15. 如权利要求 14 所述的水分散性压敏粘合带, 其特征在于含有 10-95 重量份所述微粒组分和 90-5 重量份所述水分散性聚合物组分.

16. 如权利要求 15 所述的可再浆化压敏粘合带, 其特征在于含有 50-95 重量份所述微粒组分和 50-5 重量份所述水分散性聚合物组分.

17. 如权利要求 15 所述的可洗涤压敏粘合带, 其特征在于含有 10-50 重量份所述微粒组分和 90-50 重量份所述水分散性聚合物组分.

18. 如权利要求 17 所述的可洗涤压敏粘合带, 其特征在于含有 10-40 重量份所述微粒组分和 90-60 重量份所述水分散性聚合物组分.

19. 如权利要求 14 所述的压敏粘合带, 其特征在于所述微粒组分是压敏粘合性的.

20. 如权利要求 14 所述的压敏粘合带, 其特征在于所述水分散性聚合物组分是压敏粘合性的.

21. 一种覆有如权利要求 18 所述压敏粘合带的织物.

22. 如权利要求 21 所述的织物, 其特征在于它是哺乳动物身体覆盖物形式的.

23. 如权利要求 10 所述的压敏粘合带, 其特征在于它是消毒指示带形式的.

24. 一种包括用如权利要求 14 所述压敏粘合带粘接的第一和第二再浆化带的拼接物.

25. 一种用于权利要求 1 中组分 B) 的水分散性压敏粘合剂聚合物, 它含有如下组分的乳液聚合产物:

A) 50-98 重量份聚丙烯酸烷氧基烷基酯;

B) 50-2 重量份羧酸;

C) 0-40 重量份基本上非水溶性的丙烯酸烷基酯;

D) 0-6 重量份可与所述聚丙烯酸烷氧基烷基酯或所述羧酸共聚的乳化单体.

35 26. 一种压敏粘合剂带, 其特征在于包括一层如权利要求 25 所述的通常粘着性的压敏粘合剂聚合物.

27. 如权利要求 26 所述的压敏粘合带，其特征在于所述的压敏粘合剂聚合物层涂在柔性载体的至少一个表面的至少一个部分。

28. 一种压敏粘合带，它包括柔性可再浆化的载体层、所述载体层第一表面的至少一部分上有如权利要求 25 所述的通常粘着性的压敏粘合剂聚合物层、所述载体层第二表面的至少一部分上有脱膜剂层、所述载体层的第二表面在所述载体层第一表面的反面。
5

说 明 书

压敏粘合剂及其制品

5 发明的技术领域

本发明涉及压敏粘合剂和由其制成的制品。

发明的背景

压敏粘合剂(PSA)在许多用途中得到了广泛的应用。这些粘合剂的特点是在室温(如 20 °C)下一般是粘性的，最多用很轻的指压力就可将其粘贴于表面。它们具有粘弹性和弹性的平衡，这种平衡又导致粘性、内聚性、拉伸性和弹性的四重平衡。它们具有足够的内聚性和弹性，这样它们既可被触摸又从表面上剥离，即使很粘也不会留下残余物。压敏粘合剂不能只含组合物，因为这种组合物是粘性或粘贴于表面。

15 含微粒的压敏粘合剂是一种压敏粘合剂。这种压敏粘合剂含有大量的微粒，这些微粒既可以是固有压敏粘合性的，也可以是没有压敏粘合性的，既可以是空心的，也可以是实心的。含微粒的压敏粘合剂也可含有有助于将微粒固定在衬底上的粘合剂。如有必要，这种粘合剂可以是固有压敏粘合性的。现有技术中已公开了这些压敏粘合剂的例子。例如，参见 U.S.3,857,731(Merril 等);
20 U.S.4,656,218(Kinoshita); U.S.4,735,837(Miyasaka 等); U.S.4,855,170(Darvall 等); U.S.4,994,322(Delgado 等); U.S.5,118,750(Silver 等)和 WO93/02855(Mallya 等)。这些文献中公开的粘合剂不是水分散的。再制浆时，它们会结团，并且形成粘性的小球。这些小球(在本行业中通常称作“粘性物”)会堵住再浆化方法中使用的筛子，结果增加了生产时间和费用。另外，任何不能被筛子除去的小球会影响回收利用中产生的纸产物的质量。因此，存在一种对易再浆化的水分散压敏粘性微粒粘合剂的需求。

发明的概述

30 本发明克服现有技术中的缺点。在本发明的一个方面，本发明提供一种通常为粘性的压敏粘合剂组合物，它由如下混合物构成：

- A) 不溶于溶剂但可被溶剂分散的聚合物微粒组分；
- B) 水分散的聚合物组分。

微粒组分占 0.1-99.9 重量份的粘合剂组合物。水分散的组分相应地占 99.9-0.1 重量份的粘合剂组合物。

35 在另一方面，本发明提供一种水分散的压敏粘合剂组合物，所述组合物含如下组分的乳液聚合产物：

- A) 约 50-98 重量份，最好约 80-95 重量份的聚丙烯酸烷基烷酯，以及
B) 约 50-2 重量份，最好约 20-5 重量份的羧酸。

这些主要含聚丙烯酸烷基烷酯单元的聚合物是压敏粘合性的。

本发明的粘合剂组合物最好是水分散的。与现有技术中的组合物相比，它在
5 粘着性、粘合性和耐湿性的一个或多个方面改善了性能。这种粘合剂组合物可以
制成整体形式或任何所需宽度的压敏粘合剂片材，这种片材可以窄条、带或宽的
薄膜。在任何一种情况下，压敏粘合剂片材是一个薄层(厚度一般小于 125 μ)。
另外，粘合剂层可涂于柔性基材的一个或多个相对表面。如果粘合剂层可剥离地
10 粘合于基材，则所得的产品是一种粘合剂转移带。如果粘合剂基本上永久地粘合
于基材，则该载体称为背衬。

本发明的粘合剂薄膜可用于许多用途。例如，它可用于制造可再浆化纸拼接物(永久的或临时的)。另外，这种粘合剂可用于容器的标签、消毒指示带和标签、
15 容器如信封的密封体系、外科包扎带的密封体系和哺乳动物体覆盖物(如外科手术服、被单和布帘等)。阅读了本公开书后，本发明粘合剂和带的其它用途对本行业中熟练技术人员来说是显而易见的。

在本说明书中，如下的术语具有下列意义：

“水分散的”是指颗粒材料能通过 TAPPI 试验 UM-213 或洗涤试验。以后将
详细描述所述的两个试验。

“混合”是指微粒和水分散聚合物组分的物理混合。虽然它们可通过氢键相
20 互结合，但这些组分没有被共价键合。

详细描述

微粒组分

用于本发明的微粒组分是由不溶于溶剂但可被溶剂分散的聚合物弹性体微粒
25 构成。如有必要，这些微粒可以是固有压敏粘合性的，虽然不必如此。另外，如
有必要，可加入交联剂。制造时，微粒一般是球形的。它们的平均直径一般为
1-300(最好为 1-50)微米。用于本发明的微粒可以实心的，也可以是空心的。空心
微粒可含有一个以上空隙。微粒的空隙部分的平均直径一般小于 100 微米。

微粒可用各种乳化方法制造。例如微粒可由悬浮聚合法如以下的“两步”乳
30 液聚合法制得。这种方法包括如下步骤：

a) 形成

(i) 将极性单体的水溶液与油相单体混合，形成油包水乳液，油相单体选
自(甲基)丙烯酸烷基酯和乙烯基酯；

(ii) 将油包水乳液分散到水相中，形成水包油包水(water-in-oil-in-water)乳
35 液；

b) 引发聚合，最好用加热或辐射方法。

在该方法中，第一步最好包括用亲水-亲油平衡(HLB)值低的乳化剂形成单体(至少有一些是极性单体)水溶液在油相单体(即至少一种(甲基)丙烯酸酯或乙烯基酯单体，以及任选的具有自由基反应活性的亲水低聚物和/或聚合物)中的油包水乳液。合适乳化剂的HLB值约低于7，最好为2-7。这些乳化剂的例子包括脱水山梨醇一油酸酯、脱水山梨醇三油酸酯和乙氧基化油醇如 BrijTM93(购自Atlas Chemical Industries, Inc.)。

在第一步的第一相中，将油相单体、乳化剂、自由基引发剂、和任选的具有自由基反应活性的亲水低聚物和/或聚合物以及任选的交联单体或下述的单体混合，搅拌所有或部分极性单体的水溶液，然后将其倒入油相混合物中，形成油包水乳液。可以在油相或水相中加入具有自由基反应活性的亲水低聚物和/或聚合物。油包水乳液的水相中可加入增稠剂如甲基纤维素。在第一步的第二相中，将第一步的油包水乳液分散到含HLB值约为6的乳化剂的水相中，形成水包油包水乳液。水相中也可含有任意部分的第一步中未加入的极性单体。这些乳化剂的例子包括乙氧基化脱水山梨醇一油酸酯、乙氧基化月桂醇和硫酸烷基酯。在二步中，当使用乳化剂时，它的浓度应大于它的临界胶束浓度，临界胶束浓度在本文中定义为形成胶束(乳化剂分子的亚微团粒)所需的最小乳化剂浓度。每种乳化剂的临界胶束浓度稍有不同，有用的浓度约为 1.0×10^{-4} -3.0摩尔/升。制备水包油包水乳液(即多重乳液)的其它细节可在各种参考文献中找到，如 Surfactant Systems: Their Chemistry, Pharmacy, & Biology, (D. Attwood and A. T. Florence, Chapman & Hall Limited, New York, 1983)。如果水包油包水乳液是稳定的，则会形成空心或多空隙的微粒。

该方法的第二步(最后一步)包括用热或辐射引发单体的聚合。有用的引发剂是那些一般适用于丙烯酸酯或乙烯基酯单体自由基聚合的引发剂，这些引发剂是油溶性的，在水中的溶解度非常低。然而，当极性单体是N-乙烯基吡咯烷酮时，建议将过氧化苯甲酰用作引发剂。这种引发剂包括偶氮化合物、氢过氧化物、过氧化物等以及光引发剂(如二苯酮、苯偶姻乙醚和2,2-二甲氧基-2-苯基苯乙酮)。

使用水溶性聚合引发剂会形成大量的胶乳。极小的胶乳颗粒会显著地形成不需要的胶乳。引发剂的用量一般约为0.01-10%重量，最好约为5%重量，以可聚合组份的总重量为基准。

微粒也可用较简单的乳化法(“一步法”)制备。这种方法是在至少一种乳化剂的存在下使至少一种(甲基)丙烯酸烷基酯单体或乙烯基酯单体以及任选的至少一种极性单体水相悬浮聚合。所述的乳化剂能在乳化和聚合过程中基本上稳定的微滴中产生油包水乳液。在该方法中，水悬浮液任选地和优选地还含有具有自由基反应活性亲水低聚物和/或聚合物。

在两步乳化法中，乳化剂的使用浓度大于它的临界胶束浓度。这种乳化剂的例子包括烷芳基醚硫酸盐如烷芳基醚硫酸钠如 TritonTMW/30(购自 Rohm and

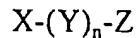
- Haas); 烷芳基聚醚硫酸盐如烷芳基聚环氧乙烷硫酸盐，最好含有高达 4 个乙氧基重复单元；以及烷基硫酸盐，如十二烷基硫酸钠、十二烷基硫酸铵、十二烷基硫酸三乙醇胺和十六烷基硫酸钠；烷基醚硫酸盐，如月桂基乙醚硫酸铵；烷基聚醚硫酸酯，如烷基聚环氧乙烷硫酸酯，最好含有高达 4 个乙氧基单元。烷基硫酸盐；
5 烷基醚硫酸盐；烷芳基醚硫酸盐及其混合物是优选的，因为它们对于最小量的表面活性剂可提供最大的空隙体积/微粒。非离子乳化剂如 SiponicTMY-500-70(乙氧基化油醇，购自 Alcolac, Inc.)和 PLURONICTMP103(一种聚环氧乙烷和聚环氧丙烷的嵌段共聚物，购自 BASF 公司)可单独使用或与阴离子乳化剂组合使用。也可加入聚合物稳定剂，但不是必需的。
- 10 “两步” 法和“一步” 法都产生单体微滴的水性悬浮液。聚合时微滴变成微粒。大多数微粒具有内部空穴。干燥时，这些空穴变成空隙。
另一种可用于制备微粒的方法形成水包油乳液，然后将此乳液分散到水相中。该水相含有至少一种(甲基)丙烯酸烷基酯单体或乙烯基酯单体、以及任选的自由基活性亲水低聚物和/或聚合物。该方法使用悬浮稳定剂，而不是乳化剂。结果，该方法形成没有内部空穴的微滴。聚合时，这些微滴变成实心微粒。
15 分离的聚合物微粒也可用美国专利 3,691,140; 4,166,152; 4,636,432; 4,656,218 和 5,045,569 中公开的悬浮聚合法制备，这些专利都描述粘合剂组合物。
通过在引发油相聚合之前，不加入所有或部分任选的自由基活性亲水低聚物和/或聚合物、和任选的极性单体，可以改进本发明微粒的制备方法。然而，在
20 100 % 转化前，必须将这些组分加入到聚合混合物。
微粒可由许多材料制成。例如，丙烯酸烷基酯和甲基丙烯酸烷基酯单体可用于制造这些微粒。这些单体是非叔醇的单官能不饱和丙烯酸酯和甲基丙烯酸酯。这些醇的烷基最好含有 4-14 个碳原子。这些丙烯酸酯单体是亲油和可水乳化的，其水溶性是有限的。如是均聚物，它的玻璃化温度一般低于 -10 °C。这种单体的
25 例子包括，但不限于如下一组单体：丙烯酸异辛酯、丙烯酸 4-甲基-2-戊酯、丙烯酸 2-甲基丁酯、丙烯酸异戊酯、丙烯酸仲丁酯、丙烯酸正丁酯、丙烯酸 2-乙基己酯、甲基丙烯酸异癸酯、丙烯酸异壬酯、丙烯酸异癸酯及其混合物。
优选的丙烯酸酯单体选自丙烯酸异辛酯、丙烯酸异壬酯、丙烯酸异戊酯、丙
30 烯酸异癸酯、丙烯酸 2-乙基己酯、丙烯酸正丁酯、丙烯酸仲丁酯及其混合物。
形成均聚物时玻璃化温度约高于 -10-0 °C 的丙烯酸酯或甲基丙烯酸酯或其它乙
烯基单体(如丙烯酸叔丁酯、丙烯酸异冰片酯、甲基丙烯酸丁酯、乙酸乙烯酯、丙
烯腈及其混合物)可任选地与一种或多种形成聚合物的玻璃化温度约低于 -10 °C
的丙烯酸酯、甲基丙烯酸酯和乙烯基酯一起使用。
适用于微粒的乙烯基酯单体包括，但不限于选自如下一组的单体：2-乙基己
35 酸乙烯酯、癸酸乙烯酯、月桂酸乙烯酯、壬酸乙烯酯、己酸乙烯酯、丙酸乙烯酯、
癸酸乙烯酯、辛酸乙烯酯以及含 1-14 个碳原子的直链或支链羧酸单官能不饱和乙

烯酯。所述的直链或支链羧酸单官能不饱和乙烯酯形成均聚物时的玻璃化温度约低于-10 °C。优选的乙烯基酯单体包括选自如下一组的单体：月桂酸乙烯酯、癸酸乙烯酯、2-乙基己酸乙烯酯及其混合物。

- 用于制备微粒的极性单体既有一点油溶性又有一点水溶性，这样可使极性单体在水相和油相之间进行分配。合适极性单体的典型例子包括，但不限于选自如下一组的单体：丙烯酸、甲基丙烯酸、衣康酸、巴豆酸、马来酸、富马酸、甲基丙烯酸磺乙酯、以及离子型单体如甲基丙烯酸钠、丙烯酸铵、丙烯酸钠、trimethylamine p-vinyl benzimide、丙酸 N,N-二甲基-N-(β -甲氧乙基)铵甜菜碱(N,N-dimethyl-N-(beta-methoxy-ethyl) ammonium propionate betaine)、N-乙烯基吡咯烷酮、N-乙烯基己内酰胺、丙烯酰胺、叔丁基丙烯酰胺、二甲氨基丙烯酰胺、N-辛基丙烯酰胺、及其混合物。优选的极性单体包括选自如下一组的单体：单烯类一元羧酸、单烯类二元羧酸、丙烯酰胺、N-取代丙烯酰胺、及其盐和混合物。这种单体的例子包括，但不限于丙烯酸、丙烯酸钠、N-乙烯基吡咯烷酮及其混合物。
- 任选地微粒中也可包括自由基活性亲水低聚物和/或聚合物。这些低聚物和/或聚合物包括，但不限于聚环氧烷烃如聚环氧乙烷、聚乙烯基甲醚、聚丙烯酰胺、聚(N-乙烯基吡咯烷酮)、聚乙烯醇、及其混合物。

微粒中所用的自由基活性亲水性低聚物和共聚物的官能化衍生物包括下列通式的大单体(macromer):

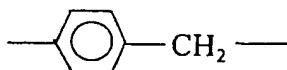
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式中

- X是可与可自由基聚合单体和极性单体自由基共聚的基团；
Y是两价连接基团；
n是0-1的整数；
Z是聚合度大于或等于2的单价亲水性聚合物或低聚物部分。

这种大单体的例子包括，但不限于，丙烯酸和甲基丙烯酸官能的低聚物和共聚物，其中X表示H₂C=CR¹-，R¹代表H或CH₃；Y是两价的羧基；n=1；Z是聚合度大于或等于2的亲水性低聚物或共聚物部分。这种大单体还包括，但不限于，对苯乙烯基官能材料，X表示H₂C=CR¹-，R¹代表H或CH₃；Y代表



- 35 n=1；Z是聚合度大于或等于2的亲水性低聚物或共聚物部分。带一个以上可与本发明微粒中所用的可自由基共聚的单体和极性单体进行自由基共聚的X基团

(这种 X 基团作为亲水性聚合物或低聚物部分 Z 的侧基或端基)的二官能或多官能低聚物和共聚物也可用在本发明的微粒中。

较好的大单体包括选自如下一组的物质：丙烯酸酯封端的聚环氧乙烷、甲基丙烯酸酯封端的聚环氧乙烷、甲氧基聚环氧乙烷甲基丙烯酸酯、丁氧基聚环氧乙烷甲基丙烯酸酯、对乙烯基苄基封端的聚环氧乙烷、丙烯酸酯封端的聚乙二醇、甲基丙烯酸酯封端的聚乙二醇、甲氧基聚乙二醇甲基丙烯酸酯、丁氧基聚乙二醇甲基丙烯酸酯，对乙烯基苄基封端的聚乙二醇、聚环氧乙烷二丙烯酸酯、聚环氧乙烷二甲基丙烯酸酯及其混合物。选择这些官能化材料的原因是由于它们能很容易地通过众所周知的离子聚合技术制得并且能高效地沿着自由基聚合的丙烯酸酯聚合物的主链提供接枝的亲水性链段。

较好的大单体还包括选自如下一组的物质：对乙烯基苄基封端的聚 N-乙烯基吡咯烷酮；对乙烯基苄基封端的聚丙烯酰胺；甲基丙烯酸酯封端的聚 N-乙烯基吡咯烷酮以及它们的混合物。正如 M.Akashi 等人的一系列论文(Angew. Makromol. Chem., 132, 81(1985); J.Appl. Polym. Sci., 39, 2027(1990); J.Polym. Sci., Part A:Polym. Chem., 27, 3521(1989))所描述的，这些大单体可通过羧基封端的 N-乙烯基吡咯烷酮或丙烯酰胺、 β -巯基丙酸链转移剂以及氯甲基苯乙烯或甲基丙烯酰氯的酯化反应制得，上述论文在本申请中参考引用。

在组合物中宜含有至少一种极性单体，但微粒还可以通过单独使用丙烯酸烷酯、甲基丙烯酸烷酯和/或乙烯基酯或仅与其它可自由基聚合的乙烯基单体(如乙酸乙烯酯)一起组合使用而制得。但是，当单独使用甲基丙烯酸酯单体时，必须加入交联剂，除非亲水性组分含有一个以上如通式 I 所定义的可自由基共聚的 X 基团。组合物中最好含有至少约 1-10 重量份的极性单体，这个比例将为微粒提供均衡的 PSA 性能。

制备微粒的组合物还可含有多官能交联剂。本申请中所用术语“多官能的”是指具有两个或多个可自由基聚合的乙烯不饱和基团的交联剂。有用的多官能交联剂包括二醇的丙烯酸酯或甲基丙烯酸酯，如二丙烯酸丁二醇酯，三元醇如丙三醇，以及四元醇如季戊四醇。其它有用的交联基包括聚合的多官能丙烯酸酯和甲基丙烯酸酯，例如聚环氧乙烷二丙烯酸酯或聚环氧乙烷二甲基丙烯酸酯；聚乙烯基交联剂，如取代或未取代的二乙烯基苯；以及二官能的氨基甲酸乙酯丙烯酸酯，如 EbecrylTM270 和 EbecrylTM230(重均分子量分别为 1500 和 5000 的丙烯酸酯化聚氨酯，均购自 Radcure Specialties)及其混合物。

以 100 重量份计，本发明所用的微粒最好含有 100 或小于 100 重量份的至少一种可自由基聚合的单体，该单体选自丙烯酸烷酯、甲基丙烯酸烷酯、乙烯基酯及其混合物；以及任选的约 0-30 重量份的一种或多种极性单体；和任选的约 0-40 重量份至少一种亲水性低聚物或聚合物组分。

以 100 重量份计，压敏微粒宜含有约 80-98 份可自由基聚合的单体，该单体选

自丙烯酸烷酯、甲基丙烯酸烷酯、乙烯基酯、及其混合物；和任选的约 1-17 份至少一种极性单体；以及任选的约 1-18 份亲水性低聚物或聚合物组分。以 100 重量份计，该压敏微粒最好含有约 87-98 份可自由基聚合的单体，和任选的约 1-8 份极性单体；以及任选的约 1-5 份亲水性低聚物或聚合物组分。

5 使用交联剂时，其用量常高达约 10 当量%。高于约 0.15 当量%(按总的可聚合微粒组合物的当量数计)，微粒就变得不粘了。给定化合物的“当量%”定义为该化合物的当量数除以在全部(微粒)组合物的总当量数，其中当量数是克数除于当量。该当量定义为分子量除于单体中可聚合的基团数(当单体仅有一个可聚合的基团时，当量 = 分子量)。可在所述微粒组合物的单体 100 % 转化成聚合物前的任何
10 时刻加入交联剂。最好在引发前加入。

上述组分的相对量对最终微粒的性能是重要的。使用较多的多官能交联剂会产生不粘的微粒。在这些的情况下，水溶性或水分散性聚合组分必须是粘性。在水溶性或水分散性的聚合组分自身是没有粘性的情况下，微粒粘结剂必须是粘性的。

15

水分散性的聚合物组分

用于本发明的聚合物组分含有足够的亲水单元，这些单元能使聚合物组分分散于水。聚合物组分最好含有能与微粒相互作用的官能团。举例来说，这些官能团包括羟基、羧基、氨基、磺酰基等。现已发现，使用含这些官能团的聚合物组分的本发明粘合剂具有良好的剪切强度。
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许多水分散性的材料可用作本发明的聚合物组分。这些材料的非限制性的例子包括选自如下一组的表面活性剂：聚环氧乙烷烷基苯基醚(如商品名称为 IGEPAL[®]CO 和 IGEPAL[®]CA，购自 Rhone-Poulenc, Inc. 的产品)；聚环氧乙烷月桂基、鲸蜡基和油基醚(如商品名称为 Brij[®]，购自 ICI Americas, Inc. 的产品)、聚环氧乙烷月桂酸酯、聚环氧乙烷油酸酯、脱水山梨醇油酸酯、环氧乙烷/环氧丙烷嵌段共聚物(如商品名称为 PLURONIC[®] 和 TETRONIC[®]，购自 BASF 公司的产品)、有机磷酸酯(如购自 International Specialty Products 的 Gafac[®] PE-510)。
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其它可用于本发明的水分散性聚合物包括 U.S.2,838,421、U.S.3,441,430、U.S.4,442,258、U.S.3,890,292、U.S.5,196,504 和 DE-C23 11 76 中公开的聚合
30 物。这些组分的例子包括，但不限于选自如下一组的物质：聚丙烯酸、聚乙烯醇、聚(N-乙烯基吡咯烷酮)、聚丙烯酰胺、聚(甲基)丙烯酸烷基烷酯(如聚丙烯酸 2-乙氧基乙酯、聚甲基丙烯酸 2-乙氧基乙酯、聚丙烯酸 2-(2-乙氧基乙氧基)乙酯和聚丙烯酸 2-甲氧基乙酯(购自 SARTOMER 公司))、聚乙烯基甲醚、聚(乙烯基甲醚：马来酸酐)(商品名称为 GantrezTM，购自 International Specialty Products 的产品)、聚醚多醇如聚丙二醇等(如商品名称为 SannixTM，购自 Sanyo Chemical Industries 的产品)、它们的共聚物等。这些物质和(甲基)丙烯酸烷基酯或乙烯基酯
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的共聚物也是适用的。如来源于秋葵和瓜耳树的树胶也可加以利用。

特别有用的水分散性聚合物含有上述的压敏乳液聚合物。当将乳液聚合物与微粒混合时，就产生剪切强度极好的压敏粘合剂组合物。这些乳液聚合物含有聚丙烯酸烷氧基烷酯和羧酸的聚合产物。另外，在聚合物组分中可加入基本上不溶于水的丙烯酸烷酯和可共聚乳化剂单体，聚合混合物一般含有 0-40 重量份，最好 0 重量份的这种丙烯酸烷酯。聚合混合物一般含有 0-6 重量份，最好 0-4 重量份的乳化剂单体。

用于制备乳液聚合物的聚丙烯酸烷氧基烷酯的例子包括，但不限于丙烯酸 2-(2-乙氧基乙氧基)乙酯、丙烯酸 2-乙氧基乙酯、丙烯酸 2-甲氧基乙氧基乙酯、甲基丙烯酸 2-甲氧基乙酯、聚乙二醇一丙烯酸酯和甲基丙烯酸酯等。

用于制备乳液聚合物的羧酸的例子包括，但不限于丙烯酸、甲基丙烯酸、衣康酸、巴豆酸、马来酸、富马酸、丙烯酸 β -羧乙酯等。

用于制备乳液聚合物的丙烯酸烷酯的例子包括，但不限于(甲基)丙烯酸甲酯、(甲基)丙烯酸乙酯、(甲基)丙烯酸正丁酯、丙烯酸 2-甲基丁酯、丙烯酸异戊酯、丙烯酸仲丁酯、丙烯酸异辛酯、丙烯酸 2-乙基己酯、丙烯酸异壬酯、丙烯酸异癸酯等。

用于制备乳液聚合物的可共聚的表面活性剂单体的例子包括，但不限于，阴离子乙烯基官能表面活性剂，如乙烯磺酸钠、苯乙烯磺酸钠、烯化聚烷氧基硫酸酯等。

可用于本发明含微粒粘合剂的另一种聚合物组分描述于未审定申请 No.08/093,080 中。该申请公开了一种含 AA、BB 和 CC 单体的压敏粘合剂聚合物。AA 单体是一种疏水性单体，选自含 2 - 14 个碳原子的丙烯酸或甲基丙烯酸非叔醇酯。该 AA 单体占 AA + BB + CC 单体的 50-80%(重量)。BB 单体选自丙烯酸 β -羧乙酯(BCEA)或其盐，以及 BCEA 或其盐的混合物，以及其他乙烯基羧酸或羧酸盐。BB 单体占 AA + BB + CC 单体的约 10-30%(重量)。如果使用 BCEA 和乙烯基羧酸或其羧酸盐，BCEA 至少占聚合物的 10 % (重量)。如果使用 BCEA 和乙烯基羧酸或其盐的混合物，BCEA 必须占聚合物的至少 10 % 重量。另外，聚合物的羧酸基团已经被碱金属的氢氧化物所中和。用于中和酸基团的氢氧化物的用量为约 0.5-2 当量的氢氧化物/酸基团。CC 单体是可分散在水中的、通式为 X-Y-Z 的大分子单体。在 CC 单体中，X 是可与 AA 和 BB 共聚的部分，Y 是连接 X 和 Z 的两价连接基团，Z 是可分散在水中的基团，Z 含有至少两个在自由基引发的共聚条件(用于形成聚合物)下基本不发生反应的单元。CC 占 AA + BB + CC 单体的 10-30%(重量)。

该压敏粘结剂在 27.5 °C，2-丁酮中的特性粘度(IV)约为 0.2 - 大于 2dl/g。较好的 IV 约为 0.7 - 1.5dl/g。最好的 IV 约为 0.9 - 1.4dl/g。

用于制备三元共聚物的 AA 单体是疏水单体丙烯酸或甲基丙烯酸非叔醇酯。所

述的非叔醇含有 2 - 14 个碳原子，最好含有 4 - 12 个碳原子。非叔醇最好是烷醇。本发明所用的术语“疏水”是指 AA 单体对水缺乏显著亲和力，即它在室温下基本上既不吸附水，也不吸收水。

适用作 A 单体的单体例子包括非叔醇的丙烯酸酯或甲基丙烯酸酯，如乙醇、

5 1-丙醇、2-丙醇、1-丁醇、2-丁醇、1-戊醇、2-戊醇、3-戊醇、2-甲基-1-丁醇、
1-己醇、2-己醇、2-甲基-1-戊醇、3-甲基-1-戊醇、2-乙基-1-丁醇、3,5,5-三甲基
-1-己醇、3-庚醇、1-辛醇、2-辛醇、异辛醇、2-乙基-1-己醇、1-癸醇、1-十二
碳醇、1-十三碳醇、1-十四碳醇等。虽然两种或多种不同的 AA 单体也是合适的，但优选的 AA 单体是丁醇或异辛醇的丙烯酸酯或其混合物。

10 AA 单体的用量较好为 50-80 % 重量，以制备三元共聚物的总的单体含量为基准。AA 单体的用量最好为 60-75 % 重量。

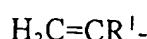
用于制备三元共聚物的 BB 单体是极性的，且可与 AA 和 CC 单体共聚。它是 BCEA 或其盐、或 BCEA 或其盐与乙烯基羧酸如丙烯酸或其盐的混合物。有用的乙烯基羧酸的例子包括丙烯酸、甲基丙烯酸、衣康酸、马来酸和富马酸。BCEA 是市售的材料。它以组分的混合物出售。例如，Rhone Poulenc, Cranbury, NJ 出售的 BCEA 是 20 % 重量的丙烯酸、40 % 重量 BCEA 和 40 % 重量其它丙烯酸低聚物的混合物。制备聚合物的 BB 单体混合物的用量最好为 10-20 % 重量。

中和剂用于制造这种聚合物。它的用量足以中和至少 50 % 的粘合剂共聚物酸部分。可以使用过量的中和剂，即高达 2 当量的中和剂/酸基。最好使用 0.75-1.5
20 当量的中和剂/酸基。中和可以使用碱金属氢氧化物或碱金属氢氧化物和少量其它中和剂的混合物。本技术领域的熟练技术人员可以理解，也可以用许多其它的中和剂。为达到所需的结果，可以改变其它中和剂的选择和用量。但是，选择的类型和用量必须能使粘合剂可被分散。最好将氢氧化钠或氢氧化钾用作中和剂。

用于制备三元共聚物的 CC 单体是水分散性的大分子单体。CC 单体最好只含
25 有一个能与 AA 和 BB 单体共聚的乙烯基。

CC 单体一般含有许多亲水部位，这些部位赋予单体所需的亲水性。

CC 单体的一种优选的 X 基团是通式 II 的乙烯基



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式中 R¹ 是氢原子或甲基。

CC 单体的一种优选的 Y 基团是二价羰基。

CC 单体的一种优选的 Z 部分是分子量为 300-30000，较好为 350-5000，更好
35 为 400-750 的低聚物/聚合物材料。Z 部分最好是聚(乙氧基噁唑啉)或聚(N-乙烯基
吡咯烷酮)或聚环氧烷烃结构。也可使用 Z 化合物，它是不同化合物如 N-乙烯基
吡咯烷酮和丙烯酰胺的共聚物。如果制备这种共聚物，只用少量(少于 10 % 重量)

的一种单体如丙烯酰.

CC 单体的用量较好占单体总重量的 10 - 30 % 重量, 更好占 15 - 25 % 重量.

本发明所用的微粒组分与水分散性组分之比可在很大的范围内变化. 例如,
5 粘合剂组合物可含 0.1-99.9 重量份的微粒组合物和 99.9-0.1 重量份的水分散性聚
合物组分. 现已发现, 这两种组分的某些较窄的范围对特定用途是优选的. 例如,
含 50-95(较好 60-95)重量份的微粒组分和 50-5(较好 40-5)重量份的水分散性聚合
物组分的粘合剂组合物可用于制备对纸拼接特别有用的带.

含 10-50(较好 10-40)重量份微粒组分和 90-50(较好 90-40)重量份水分散性聚合
10 物组分的粘合剂组合物在外科包扎带的密封体系和哺乳动物体覆盖物(如外科手
术服、被单和布帘等)等方面特别有用. 在这些用途中, 可以使用这种带, 而且在
消毒过程中不会失去压敏粘合性. 也可同时使用消毒指示剂, 以指示捆扎的包裹
已通过消毒周期. 然而, 可以洗涤使用本发明粘合剂的包装物和带, 因为本发明
的粘合剂分散在碱性洗涤溶液中. 因此, 洗涤前不必除去粘合剂.

15 改性剂

本发明的粘合剂组合物可任选地含有一种或多种改性剂, 以赋予或提高与制
备粘合剂或与其应用有关的所需性质. 改性剂的用量一般足以产生所需的效果,
但不影响粘合剂的压敏粘合性. 有用改性剂的例子包括, 但不限于增稠剂如选自
20 碱溶胀缩合聚合物的增稠剂, 如名称为 UCAR® POLYPHOBE®, 购自 Union Carbide,
Inc. 的商品; Alcogum™(购自 Alco Chemical); Rheolate™(购自 Rheox);
Acrysol™(购自 Rohm & Haas); 和 Carbopol™(购自 B. F. Goodrich).

本发明中也可使用通常与增稠剂或羧酸官能聚合物组合使用的中和剂. 这些
中和剂包括, 但不限于金属离子, 如钠、钾、钙、镁、铜、铝或锌; 以及胺, 如
氨和有机胺如正己胺、单乙醇胺和己二胺.

25 可用于改变粘合剂组合物性质的其它改性剂包括, 但不限于增粘树脂、增塑
剂、颜料、填料、流动控制剂和稳定剂.

用作本发明制品柔性载体或背衬的合适材料包括, 但不限于纸、胶乳饱和纸、
聚合物膜、乙酸纤维素膜、乙基纤维素膜、布(即由合成或天然材料制成的织造或
非织造片材)、金属箔和陶瓷薄板.

30 可用作柔性载体的材料的例子包括聚烯烃(如聚乙烯、聚丙烯(包括全同立构的
聚丙烯)、聚苯乙烯、聚酯、聚乙烯醇、聚对苯二甲酸乙二醇酯、聚对苯二甲酸丁
二醇酯、聚己内酰胺、聚(1,2-二氟乙烯)等)等. 用于本发明的市售背衬材料包括
牛皮纸(购自 Monadnock Paper, Inc.); 玻璃纸(购自 Flexel Corp.); 纺时粘接(spun-
bond)聚乙烯和聚丙烯, 如 Tyvek™和 Typar™(购自 DuPont, Inc.); 以及由聚乙烯
35 和聚丙烯制得的多孔膜, 如 Teslin™(购自 PPG Industries, Inc.)和 Cellguard™(购自
Hoechst-Celanese).

柔性载体也可包括涂有脱膜剂的衬底。制造粘合剂转移带时一般使用这种衬底。涂有脱膜剂的衬底在本行业中是众所周知的。举例来说，它们包括涂有聚硅氧烷的牛皮纸等。

5 本发明的带也可包括低粘性的背面涂胶(LAB)。这种 LAB 一般涂在带的压敏粘合剂表面的背面。LAB 在本行业中是已知的。

用如下的实施例说明本发明的这些或那些实施方案，这些实施例不应看作是对本发明范围的限制。制备本发明的实施例，然后用如下的试验方法检测其耐洗涤性、粘着性、粘合性、剪切能力和再浆化能力。

10 试验方法

粘着性

15 在该试验中，将一个直径为 8.13 cm、轮宽为 1.9 cm 的 Delrin 轮(重为 29 克)沿一个长度为 26.7cm、与水平面夹角为 24° 的平面滚下，在该水平面上放置需测试的带，粘合剂面向上。测量该轮沿水平粘合剂表面滚动的距离，粘着性与轮滚动的距离成反比。

再浆化能力

再浆化能力是按 TAPPI 试验 UM-213 测量的。对于双面带，将一张 20 cm × 2.54 cm 的带条夹在两张 20 cm × 2.54 cm 吸墨纸条间。对于单面带，将两张 20 cm × 2.54 cm 的带条贴在吸墨纸上。将试样切成约 1.5 cm² 方块。然后将足够数目的 1.5 cm² 吸墨纸片加入到带/吸墨纸粘合片中，使试样的总重达到 15 克。然后将试样放入装有 500 ml 室温自来水的韦林氏掺合器中。掺合器转动 20 秒钟后，停 1 分钟，用水壶将溅到掺合器边上和盖上的纸料冲洗到底部。然后将掺合器再转动 20 秒钟，按前述的方法将纸料再冲回底部后，掺合器再转动最后 20 秒钟。从掺合器中取出纸料，在纸巾模子中制成纸巾。从模子中取出纸巾，将其放在吸墨纸间在水压机中压 90 秒钟，然后干燥，检验任何未再浆化带的颗粒。如果只有 2 个或很少几个颗粒，则认为，该带通过试验。存在一个或二个颗粒不构成失败，因为它们可能来自污染的设备或筛网。当材料不能通过试验时，颗粒会被分散整个纸巾中。

30 上述的试验不适用于背衬是不能再浆化的材料如聚酯薄膜的带产品。这种背衬上所用的粘合剂的分散能力可用测试粘合剂转移带或水分散性载体层上粘合剂膜的方法加入测量。

洗涤试验

35 将一片需测试的带层压到未处理的 50 % 棉/50 % 聚酯织物(购自 Standard Textile Co., Cincinnati, OH，商品名称为 Barrier Supreme™)上，制成试样。用 1.8

kg(4lb)橡胶轮将该带层压到该织物上,使其露出含微粒的粘合剂.然后用 134 °C, 10 分钟的四脉冲预真空循环和 1 分钟蒸汽干燥对试样进行蒸汽消毒.

然后将试样放在 60 lb. 的市售洗衣机 (Milnor washer, Model No. 36021 BWE/AEA; Pillerin Milnor Corp., Kenner, LA) 中进行洗涤. 使该试样经受一般的外科亚麻布洗涤循环. 该洗涤循环包括: (a) 在 0.1% Paralate 55 GL11™ 市售液体洗涤碱(Ecolab Inc., St. Paul, MN)中冷碱洗涤 3 分钟; 3 分钟冷水冲洗, 用 0.1% Paralate 55 GL11™ 市售液体洗涤碱(Ecolab Inc., St. Paul, MN) 和 0.05% Kindet™ 市售洗涤剂(Ecolab Inc.)8 分钟热 break/suds 洗涤; 二个 3 分钟热水冲洗; 一个 3 分钟冷水冲洗; 用 0.05% Tri Liquid sour 55GL™ 市售液体洗涤酸(sour)(Ecolab Inc.) 和 0.05% Tex Special Liquid™ 市售液体衣服润滑剂(Ecolab Inc.)3 分钟冷的酸/软漂清; 6 分钟压榨, 除去过量的液体.

检查每个试样, 通过试验是指织物没有粘合剂的残余物, 而没有通过试验是指织物上有粘合剂残余物.

15

粘合性

本试验是对纸的 180° 剥离粘合性. 用双面压敏粘合带将 3.175 cm 高级书写纸条(bond paper)层压到 5 cm × 12.5 cm 不锈钢测试板上. 然后将 2.54 cm × 25 cm 试样带条的一端覆盖在层压的高级书写纸上(如试样是双面带, 则将 68 g/m² 牛皮纸背衬放在试样带的一个粘合剂面上). 用 2 kg 滚轮以 30.6 mm/min 的速度滚过 12.5 cm 长的试样, 以确保粘合剂接触均匀. 然后将试样向自己一面折, 然后以 30.5 cm/min 的速度从高级书写纸的表面剥离. 用在 21 °C 和 50 % 相对湿度下工作的 Instron Model 1122 记录剥离带所用的力.

剪切强度

使用 2.54 cm × 15 cm 带条的 2.54 cm × 2.54 cm 端部. 如果该带只在一个表面有粘合剂, 则将粘合剂面贴在光亮的退火钢测试板上, 并用 2kg 滚轮滚两遍. 如果该带在每一个表面上有粘合剂, 则将需测试剪切强度的粘合剂贴在光亮的退火钢板上, 然后将一片 68g/m² 特别光洁的牛皮纸背衬层压到另一粘合剂面. 然后将钢板夹在一个与垂直方向偏 2° 夹具上. 这样使带的游离端以与测试板相差 178° 的角度向下延伸, 在该端上系上一个 1000 g 的重物. 记录带从测试板上分离的时间(近似为分钟), 剪切强度与花费的时间直接相关. 其它的细节参见上述的压敏粘合带的测试方法、试验 PSTC-7. 对于用本发明粘合剂制成的带, 需有尽可能长的剪切时间, 较好超过 1000 分钟, 虽然剪切时间低至 40 分钟, 粘合剂也是令人满意的.

35

缩写

在本申请公开的实施例中使用如下缩写.

AA = 丙烯酸

BA = 丙烯酸丁酯

BCEA = 丙烯酸 β -羧乙酯

5 PEOA = 聚环氧乙烷一丙烯酸酯, 平均分子量为 750

EOEA = 丙烯酸 2-乙氧基乙酯

EOEOEA = 丙烯酸 2-(2-乙氧基)乙氧基乙酯

HDDH = 1,6-己二醇二丙烯酸酯

IOA = 丙烯酸异辛酯

10 MSA = 微粒粘合剂

PC = 水分散性聚合物组分

PPG = 聚丙二醇

PAA = 聚丙烯酸

PVA = 聚乙烯醇

15 Repulp = 再浆化试验

P = 通过(再浆化试验)

F = 没有通过(再浆化试验)

每个制备开头处括号中的内容表示制备中所用的单体组分和每个组分的重量百分数.

20

微粒的制备

微粒组分 A(IOA:AA:PEOA/97:2:1)

将 4.8 gAA、2.4 g PEOA 和 1.13g LucidolTM -70(70 % 过氧苯甲酰, 购自 Elf Atochem)溶解在 232 g IOA 中. 将 0.75 g SiponateTMMDS-10(十二烷基苯磺酸钠表面活性剂, 购自 Rhone-Poulenc)溶解于 360g 水. 然后把 IOA 混合物加入到表面活性剂溶液中, 用 OmniTM混合机乳化, 直到液滴尺寸小于 5 微米. 然后将含固量为 40 % 的乳液加入到 1 升折流(baffled)反应器中, 加热到 65 °C, 用氮气脱气, 让其反应 8 小时.

30 微粒组分 B(IOA:AA:PEOA/89:1:10)

将 2.1g AA、21 gPEOA、0.21 g 聚(环氧乙烷), 二甲基丙烯酸酯和 0.99 g LucidolTM70 溶解于 189.5 gIOA 中, 将 6 g StandapolTM(一种表面活性剂, 即十二烷基硫酸铵, Hercules)溶解于 390 g 水中. 然后把 IOA 混合物加入到表面活性剂溶液中, 用 OmniTM混合机乳化, 直到液滴尺寸小于 5 微米. 然后将乳液加入到 1 升压陷(indented)树脂瓶中, 加热到 65 °C, 用氩气脱气, 让其反应 18 小时.

微粒组分 C(IOA:BA:AA:PEOA/79:17:1:3)

将 2.1 gAA、8.4 g PEOA、39.9 g BA、0.25 g HDDA 和 0.99 g Lucidol™-70 溶解于 189.9 g IOA 中，将 6.5 g Standapol™(一种表面活性剂，即十二烷基硫酸铵，Hercules)溶解于 390 g 水中。然后把 IOA 混合物加入到表面活性剂溶液中，
5 用 Omni™混合机乳化，直到液滴尺寸小于 5 微米。然后将乳液加入到 1 升压陷树脂瓶中，加热到 65 °C，用氩气脱气，让其反应 22 小时。

微粒组分 D(IOA:AA:HDDA/96:2:2)

将 4.2 gAA、4.2 g HDDA 和 1.13 g Lucidol™-70 溶解于 230g IOA 中，将 0.75
10 g 十二烷基苯磺酸钠表面活性剂溶解于 360g 水中。然后把 IOA 混合物加入到表面活性剂溶液中，用 Omni™混合机乳化，直到液滴尺寸小于 5 微米。然后将乳液加入到 1 升压陷树脂瓶中，加热到 65 °C，用氩气脱气，让其反应 12 小时。所得微粒是非粘性的。

15 微粒组分 E(100%IOA)

在一个装有 450 ml 去离子水和 4 g Standapol™ 的 1 升压陷树脂瓶中制备微粒。将该水溶液以 400 rpm 的速度搅拌，加热到 70 °C，用氩气脱气。在热的表面活性剂水溶液中加入 150 g IOA 和 0.71 g Lucidol™。然后将温度降到 65 °C，让混合物反应 22 小时。经用光学显微镜测量，平均粒径约为 33 微米。
20

水分散性聚合物组分的制备

PC-1(EOESEA:AA/80:20)

在装有冷凝器、搅拌器和氮气吹扫管的 3 升烧瓶中加入 1217 g 去离子水和 0.39 g 过硫酸钾。将混合物加热至 76 °C，用氮气吹扫，并以 150 rpm 的速度搅拌。制备进料 I、II 和 III。进料 I 含有溶解于 100g 去离子水的 13.5 g Mazon™ SAM-211(购自 PPG/Mazer Chemicals)。进料 II 含有溶解于 50 去离子水的 0.39 g 过硫酸钾。进料 III 含有 240 g EOESEA、60g AA 和 0.09 g 叔十二烷基硫醇。将进料 I 和 75.0 g 进料 III 加入烧瓶中，反应 30 分钟。然后将其余部分的进料 III 和所有的进料 II 在 1-2.5 小时内逐渐加入到烧瓶中。加入所有的进料后，再让烧瓶中的内容物反应 35 分钟。让乳化的内容物冷却，并从烧瓶中将其倒出。它的 pH = 3.32，布洛克菲尔德粘度=5 cps，粒度为 182μ。
30

PC-2(EOESEA:AA/85:15)

在装有冷凝器、搅拌器和氮气吹扫管的 2 升烧瓶中加入 800 g 去离子水和 0.39 g 过硫酸钾。将混合物加热至 76 °C，用氮气吹扫，并以 300 rpm 的速度搅拌。制备进料 I、II 和 III。进料 I 含有溶解于 100g 去离子水的 13.5 g Mazon™ SAM-

211. 进料 II 含有溶解于 50 去离子水的 0.39 g 过硫酸钾. 进料 III 含有 255 g EOEOEA、45 g 和 0.09 g 叔十二烷基硫醇. 将进料 I 和 75.0 g 进料 III 加入烧瓶中, 反应 30 分钟. 然后将其余部分的进料 III 和所有的进料 II 在 1-2.5 小时内逐渐加入到烧瓶中. 加入所有的进料后, 再让烧瓶中的内容物反应 35 分钟. 让乳化的内容物冷却, 并从烧瓶中将其倒出. 聚合物的粒度为 94.3 μ .

5 PC-3(EOEOEA:BA:AA/75:15:10)

在装有冷凝器、搅拌器和氮气吹扫管的 2 升烧瓶中加入 800 g 去离子水和 0.39 g 过硫酸钾. 将混合物加热至 76 °C, 用氮气吹扫, 并以 300 rpm 的速度搅拌. 制备进料 I、II 和 III. 进料 I 含有溶解于 100g 去离子水的 13.5 g Mazon™ SAM-211. 进料 II 含有溶解于 50 去离子水的 0.39 g 过硫酸钾. 进料 III 含有 255 g EOEOEA、45 g BA、30 g AA 和 0.09 g 叔十二烷基硫醇. 将进料 I 和 75.0 g 进料 III 加入烧瓶中, 反应 30 分钟. 然后将其余部分的进料 III 和所有的进料 II 在 1-2.5 小时内逐渐加入到烧瓶中. 加入所有的进料后, 再让烧瓶中的内容物反应 35 分钟. 让乳化的内容物冷却, 并从烧瓶中将其倒出. 聚合物的粒度为 236 μ .

15 PC-4(EOEOEA:BA:AA/70:15:15)

在装有冷凝器、搅拌器和氮气吹扫管的 3 升烧瓶中加入 1672 g 去离子水、20.9g Mazon™ SAM-211 和 2.1 g 过硫酸钾. 将混合物加热至 76 °C, 用氮气吹扫, 并以 200 rpm 的速度搅拌. 制备含 292.6 g EOEOEA、62.7 g BA、62.7 g AA 和 0.30 g 叔十二烷基硫醇的预混合物. 用滴液漏斗在 5 小时内将该预混合物加入到烧瓶中. 让乳化的内容物冷却, 并从烧瓶中将其倒出. 它的 pH = 2.46, 布洛克菲尔德粘度=4.5 cps, 粒度为 108 μ .

25 PC-5(EOEOEA:BA:AA/50:35:15)

在装有冷凝器、搅拌器和氮气吹扫管的 3 升烧瓶中加入 1672 g 去离子水、20.9g Mazon™ SAM-211 和 2.1 g 过硫酸钾. 将混合物加热至 76 °C, 用氮气吹扫, 并以 200 rpm 的速度搅拌. 制备含 209 g EOEOEA、146.3 g BA、62.7 g AA 和 0.21 g 叔十二烷基硫醇的预混合物. 用滴液漏斗在 6 小时内将该预混合物加入到烧瓶中. 让乳化的内容物冷却, 并从烧瓶中将其倒出. 它的 pH = 2.55, 布洛克菲尔德粘度=4.5 cps, 粒度为 91.0 μ .

PC-6(EOEOEA:BCEA/82:18)

在装有冷凝器、搅拌器和氮气吹扫管的 3 升烧瓶中加入 2000 g 去离子水和 0.65 g 过硫酸钾. 将混合物加热至 76 °C, 用氮气吹扫, 并以 150 rpm 的速度搅拌. 制备进料 I、II 和 III. 进料 I 含有溶解于 100g 去离子水的 22.5 g Mazon™ SAM-

211. 进料 II 含有溶解于 50 去离子水的 0.65 g 过硫酸钾. 进料 III 含有 410 g EOEOEA、90 g BCEA 和 0.15 g 叔十二烷基硫醇. 将进料 I 和 75.0 g 进料 III 加入烧瓶中, 反应 30 分钟. 然后将其余部分的进料 III 和所有的进料 II 在 1-5 小时内逐渐加入到烧瓶中. 加入所有的进料后, 再让烧瓶中的内容物反应 35 分钟.
- 5 让乳化的内容物冷却, 并从烧瓶中将其倒出. 它的 pH = 4.47, 布洛克菲尔德粘度=1.5 cps, 粒度为 146 μ .

PC-7(EOEOEA:BA:AA/60:20:20)

在装有冷凝器、搅拌器和氮气吹扫管的 3 升烧瓶中加入 1217 g 去离子水和 0.39 g 过硫酸钾. 将混合物加热至 76 °C, 用氮气吹扫, 并以 150 rpm 的速度搅拌. 制备进料 I、II 和 III. 进料 I 含有溶解于 100g 去离子水的 13.5 g Mazon™ SAM-211. 进料 II 含有溶解于 50 去离子水的 0.39 g 过硫酸钾. 进料 III 含有 180 g EOEOEA、60g BA 和 0.09 g 叔十二烷基硫醇. 将进料 I 和 75.0 g 进料 III 加入烧瓶中, 反应 30 分钟. 然后将其余部分的进料 III 和所有的进料 II 在 1-2.5 小时内逐渐加入到烧瓶中. 加入所有的进料后, 再让烧瓶中的内容物反应 30 分钟. 让乳化的内容物冷却, 并从烧瓶中将其倒出. 乳化反应产物的粒度为 101 μ , 布洛克菲尔德粘度=5 cps.

PC-8(EOEOEA:IOA: β -CEA/50:17:33)

20 在装有冷凝器、搅拌器和氮气吹扫管的 3 升烧瓶中加入 1200 g 去离子水和 0.39 g 过硫酸钾. 将混合物加热至 76 °C, 用氮气吹扫, 并以 150 rpm 的速度搅拌. 制备进料 I、II 和 III. 进料 I 含有溶解于 100g 去离子水的 13.5 g Mazon™ SAM-211. 进料 II 含有溶解于 50 去离子水的 0.39 g 过硫酸钾. 进料 III 含有 150 g EOEOEA、51.0 g IOA、99.0 g BECE 和 0.09 g 叔十二烷基硫醇. 将进料 I 和 75.0 g 进料 III 加入烧瓶中, 反应 30 分钟. 然后将其余部分的进料 III 和所有的进料 II 在 1-3 小时内逐渐加入到烧瓶中. 加入所有的进料后, 再让烧瓶中的内容物反应 35 分钟. 让乳化的内容物冷却, 并从烧瓶中将其倒出. 乳化反应产物的 pH=3.32, 布洛克菲尔德粘度=3 cps, 粒度为 201 μ .

30 PC-9 (EOEOEA:AA/95:5)

在装有冷凝器、搅拌器和氮气吹扫管的 2 升分瓣(split)树脂瓶中加入 800 g 去离子水和 0.39 g 过硫酸钾. 将混合物加热至 76 °C, 并以 150 rpm 的速度搅拌. 以每分钟 1 升的氮气吹扫制备进料 I、II 和 III. 进料 I 含有溶解于 100g 去离子水的 15.0 g Mazon™ SAM-211. 进料 II 含有溶解于 50 去离子水的 0.39 g 过硫酸钾. 35 进料 III 含有 285 g EOEOEA(购自 Sartomer Chemical Co.)、15.0 g AA 和 0.09 g 叔十二烷基硫醇. 将进料 I 和 75.0 g 进料 III 加入烧瓶中, 反应 30 分钟. 然后将

其余部分的进料 III 和所有的进料 II 在 1-2.5 小时内加入到烧瓶中。再让烧瓶中的内容物反应 30 分钟。然后让乳化的内容物冷却，并从烧瓶中将其倒出。

PC-10 (IOA:AA/85:15)

5 在装有冷凝器、搅拌器和氮气吹扫管的 3 升分瓣树脂瓶中加入 800 g 去离子水和 0.39 g 过硫酸钾。将混合物加热至 76 °C，用氮气(1 l/min)吹扫，并以 150 rpm 的速度搅拌。制备进料 I 和 II。进料 I 含有溶解于 100g 去离子水的 13.5 g Mazon™ SAM-211。进料 II 含有 225 g IOA、45.0 g AA 和 0.09 g 叔十二烷基硫醇。将进料 I 和 75.0 g 进料 II 加入烧瓶中，并让其在 76 °C 反应 30 分钟。然后在 2 小时内
10 加入其余部分的进料 II。然后再让烧瓶中的内容物反应 30 分钟。让所得乳化反应产物冷却，并从烧瓶中将其倒出。乳化反应产物的固体含量 26.8% 重量，粒度为 128nm。

PC-11(EOEOEA:AA/90:10)

15 在装有冷凝器、搅拌器和氮气吹扫管的 5 升烧瓶中加入 2403 g 去离子水和 1.17 g 过硫酸钾。将混合物加热至 76 °C，用氮气吹扫，并以 150 rpm 的速度搅拌。制备进料 I、II 和 III。进料 I 含有溶解于 297.0 g 去离子水的 40.5 g Mazon™ SAM-211。进料 II 含有溶解于 100 去离子水的 1.17 g 过硫酸钾。进料 III 含有 810 g EOEOEA、90.0 g AA 和 0.27 g 叔十二烷基硫醇。将进料 I 和 300 g 进料 III 加入烧瓶中，反应 30 分钟。然后将其余部分的进料 III 和所有的进料 II 在 1-2.5 小时内逐渐加入到烧瓶中。然后，再让烧瓶中的内容物反应 30 分钟。让乳化的反应产物冷却，并从烧瓶中将其倒出。

PC-12 (EOEOEA:AA/90:10)

25 在 25 加仑(95 l)搪瓷反应器中加入 62.3 kg 去离子水和 21.2 g 过硫酸钾，将其加热至 76 °C。将反应器中的内容物以 75 rpm 的速度搅拌，并用氮气吹扫。制备进料 I 和 II。进料 I 含有 1.6 kg AA、735 g Mazon™ SAM-211、14.7 kg EOEOEA 和 4.9 g 叔十二烷基硫醇。进料 II 含有 5.3 kg 去离子水和 21.2 过硫酸钾。将 4.3 kg
30 进料 I 加入烧瓶中，并让其反应 30 分钟。然后在 1.5-2 小时内加入其余部分的进料 I 和所有进料 II。然后再让烧瓶中的内容物反应 1 小时。让所得乳化反应产物冷却，并从烧瓶中将其倒出。乳化反应产物的粒度为 228 μ ，布洛克菲尔德粘度 =4 cps。

实施例 1-10

35 通过混合微粒与 1 % 重量结合的(associative)碱溶胀增稠剂 (UCAR Polyphobe™104，购自 Union Carbide)制备仅使用微粒组分 A-E 的粘合带试样，

然后用氢氧化钾和聚(氧化丙烯)叔胺中和到 pH 等于 7。将粘合剂涂在脱膜表面 (release surface)，然后在 79 °C 的烘箱中干燥 3 分钟，将层压到“Crystex”薄纸上，得到单面结构。为得到双面结构，将粘合剂涂在单面试样的反面，并按相似方法干燥。剥离脱膜衬里后，进行测试。

- 5 按如下方法制备实施例 1-10 的粘合带试样：将微粒组分 A 与所需水溶性或水分散性的聚合物组分混合，然后按上述由微粒组分 A-E 制成的带所用的相同方法增稠、中和和涂布。所用水溶性或水分散性的聚合物组分是购自 International Specialty Products 的有机磷酸酯 Gafac® PE-510、分子量为 8×10^5 g/mol 的聚乙
10 烯醇 PVA、分子量为 2×10^5 g/mol 的聚丙烯酸 PAA、分子量为 10^6 g/mol 的聚环
氧乙烷 PEO、购自 International Specialty Products 的乙氧基化烷基酚 Igепал®
CA-520、CA-630 和 C-630、购自 Sanyo Chemical Industries 的聚亚烷基二醇
Sannix® SP-750、和分子量为 400 g/mol 的聚丙二醇 PPG。

这些实施例表明：在微粒粘合剂中加入水溶性或水分散性的聚合物组分可以提高粘性，而且仍能保持再浆化能力。这些结果列于表 1 中。

15

表 1

实施例	聚合物组分	% P.C.	粘性 (mm)	再浆化试验 (P/F)
对比	无(全部微粒组分 A)	0	80	P
1	Gafac®PE-510	0.5	15	P
2	Gafac®PE-510	1.0	20	P
3	PVA	1.0	33	P
4	PAA	1.0	38	P
5	PEO	1.0	29	P
6	Igepal®CA520	2.5	48	P
7	Igepal®CA630	2.5	47	P
8	Igepal®CO630	2.5	36	P
9	Sannix®SP750	5.0	30	P
10	PPG	5.0	33	P

实施例 11-21

用微粒组分 A 和表 2 所列的聚合物组分按实施例 1-10 所述的相同的方法制备实施例 11-21。但不加增稠剂。

20

这些实施例表明：当加入的水溶性或水分散性聚合物组分是 PSA 时，所有情况下粘合性能都有提高，因为在绝大多数情况下是有粘着性，而且能保持再浆化能力。结果列于表 2 中。

表 2

实施例	聚合物组分(PC)	% PC 组分	粘着性 (mm)	再浆化试 验(P/F)	粘合性 (N/cm)
对比	无(所有微粒组分 A)	0	80	P	3.6
11	PC-1	22	56	P	6.9
12	PC-2	22	17	P	8.0
13	PC-3	22	20	P	7.3
14	PC-4	22	123	P	7.1
15	PC-5	22	79	P	6.9
16	PC-6	22	14	P	8.4
17	PC-7	13	38	P	6.3
18	PC-8	13	20	P	7.1
19	PC-1	13	33	P	6.4
20	PC-9	13	21	P	5.8
21	PC-10	22	32	P	6.0

实施例 22-33

在实施例 22-33 中, 所用的水溶性或水分散性的聚合物组分是 PC-11, 微粒组分是微粒组分 A。实施例 22 按实施例 1 相同的方法进行制备。实施例 23-32 按实施例 11 相同的方法进行制备。实施例 33 按实施例 11 所述的方法进行中和。

这些实施例在宽的聚合物组分浓度范围内显示了本发明的性质。如表 3 所示这些结果显示与对比例 A 相比, 提高了的粘着性能、粘合性能和剪切性能(在绝大多数情况), 同时仍保持再浆化能力。

表 3

实施例	聚合物组分(%)	粘着性 (mm)	再浆化试 验(P/F)	粘合性 (N/cm)	室温剪切粘 合性(分)
对比例 A	0(所有微粒组分 A)	80	P	3.6	1330
22	5	62	P	5.4	2800+
23	92	37	P	5.4	2800+
24	13	24	P	5.4	2800+
25	23	21	P	6.1	2800+
26	31	18	P	6.1	2800+
27	38	18	P	5.7	2800+
28	43	22	P	6.1	2020(剪切)
29	47	22	P	6.5	1650(剪切)
30	60	35	P	5.7	130(剪切)
31	75	33	P	5.9	76(剪切)
32	90	39	P	5.9	57(剪切)
33	100(所有 PC-11)	31	P	7.8	130(剪切)

对比例 A 在 1330 分钟后突然掉下. 实施例 28、29 和 33 在所示的时间切变.

5

实施例 34-38

这些实施例表明了将由不同比例的 EOEOEA 和丙烯酸共聚物构成的水分散性聚合物组分与不同类型微粒粘合剂混合物的作用. 实施例 34、35、36、37 和 38(含聚合物组分)是按实施例 11 相同的方法制备的.

10 对比例 B 和实施例 34 比较含 10 % PEOA 的微粒组合物. 对比例 B 不用聚合物组分. 实施例 34 用 22 % 重量 PC-5. 如表 4 所示, 加入 PC-5 提高了粘合性和再浆化能力.

表 4

实施例	微粒组分	PC 组分	粘着性 (mm)	再浆化试验 (P/F)	粘合性 (N/cm)
对比例 B	B	...	19	F	5.8
34	B	PC-5	35	P	6.7

15

对比例 C 和实施例 35 比较含 PEOA 和丙烯酸丁酯的微粒组合物. 对比例 C 不用聚合物组分. 实施例 35 含 22 % 重量 PC-1. 如表 5 所示, 加入 PC-1 提高了粘

合性和再浆化能力.

表 5

实施例	微粒组分	PC 组分	粘着性 (mm)	再浆化试 验(P/F)	粘合性 (N/cm)
对比例 C	C	...	33	F	3.7
35	C	PC-1	180	P	5.6

对比例 D 和实施例 36 和 37 比较无粘着性的微粒组合物. 对比例 D 不含聚合
5 物组分. 实施例 36 和 37 分别使用 33 和 43 % 重量的 PC-4. 如表 6 所示, 加入
PC-4 提高了粘着性和粘合性, 同时仍保持再浆化能力.

表 6

实施例	微粒组分	PC 组分	粘着性 (mm)	再浆化试 验(P/F)	粘合性 (N/cm)
对比例 D	D	...	***	P	***
36	D	PC-4	150	P	1.3
37	D	PC-4	150	P	2.9

***无粘着性, 不能测试

10

相对于本申请公开的所有其它实施例(直约为 3 微米), 对比例 E 和实施例 38
15 比较粒度较大的(直径为 33 微米)微粒组合物. 对比例 E 不用聚合物组分. 实施例
38 使用 43 % 重量 PC-12. 加入 PC-12 提高了粘着性和再浆化能力.

15

表 7

实施例	微粒组分	PC 组分	粘着性 (mm)	再浆化试 验(P/F)	粘合性 (N/cm)
对比例 E	E	...	170	F	6.1
38	E	PC-12	130	P	4.1

实施例 39-42

制备本发明的压敏粘合带, 将其层压在未处理的 50 % 棉/50 % 聚酯织物(Barrier
Supreme™), 然后按所述的洗涤试验方法进行洗涤. 带中所用的背衬是“Crystex”
20 薄纸. 表 8 中列出了测试的粘合剂组合物和所得结果.

表 8

实施例	微粒组分		水分散性组分	
	Mat'l.	重量份	Mat'l.	重量份
39	A	78	PC-6	22
40	A	87	PC-7	13
41	A	87	PC-1	13
42	A	87	PC-8	13

所有的试样在洗涤织物上没有显示残余物的证据。

实施例 43

5 制备水分散性的聚合物。经费歇尔酯化 CarbowaxTM750(Union Carbide)制备分子量为 750 的单甲氧基聚乙二醇丙烯酸酯单体。将 100 份 CarbowaxTM750 和 100 份甲苯加入装有迪安·斯达克冷凝器和搅拌器的烧瓶中。将该溶液在回流温度加热 2 小时。向该混合物中加入 11.3 份丙烯酸、4.5 份对甲苯磺酸、3000 ppm IrganoxTM PS 800(Ciba-Geigy)和 500 ppm 吡噻嗪。再将此溶液在回流温度加热 16 小时。冷却至室温时，用 4.5 份氢氧化钙中和过量的酸。将形成的沉淀过滤掉。然后减压蒸去甲苯，得到固态的 100 % 丙烯酸酯单体。

10 水分散性压敏粘合剂聚合物的制备方法是：将丙烯酸丁酯(BA)、BCEA(Rhone Poulenc 出售的 20 % 重量丙烯酸、40 % BCEA 和 40 % 重量其它丙烯酸低聚物的混合物)、单甲氧基聚乙二醇丙烯酸酯单体加入到装有乙酸乙酯、甲醇、异丙醇和 15 0.51 g 偶氮二异丁腈溶剂混合物的 1 夸脱瓶中，该单体进料含有 32-34 % 聚合混合物。以 1 升/分的速度用氮气吹扫 2 分钟，使该混合物脱氧。将该瓶密封，并在 55 °C 旋转水浴中放置 24 小时，使其基本上完全聚合。然后在 27.5 °C 的 2-丁酮中测量所得聚合物的特性粘度(IV)。向该粘合剂聚合物中掺入溶于 3.57N 50 : 50 甲醇：水(V/V)溶液的中和剂(86.8%纯 KOH，按 100 纯 KOH 计，1.25 中和当量) 20 然后将微粒(A)混入粘合剂聚合物中，得到 20g 微粒对 100g 粘合剂聚合物的最后比率(即 16.7% 重量的微粒)。

25 将所得中和的含微粒的压敏粘合剂组合物涂在 29 磅基重牛皮纸上(Mosinee Paper Corporation of Mosinee, Wisconsin 制造的 M-2383 Smooth Crepe Semi-Bleached Kraft Saturating Paper)，形成下述的密封带。

按 0.68 g/154.8 cm²(24in²)的用量用标准的实验室刮涂器将溶于溶剂体系中的压敏粘合剂涂布在牛皮纸上，然后在 100 °C 的强制烘箱中干燥 15 分钟。

表 9 列出了涂在氟化学处理的聚酯包装纸上的实施例 43 和对比例 1 密封带的初始粘合性、消毒后粘合性、消毒前包装纸密封(T(0))、消毒后包装纸密封(T(A))和分散性。使用如下的测试方法。

对处理过的帷帘包装物的初始粘合性

将氟化学处理过的聚酯帷帘(购自 Standard Textile Co., Cincinnati, OH as WrapPel™)试样切成 3.81 cm × 10.16 cm 带，并用 # 410 双面涂布带(购自 3M 公司)将其粘贴在 5.08 cm × 12.2 cm 钢板上。将本发明的实例密封带(2.54 cm × 10.16 cm)贴在该帷帘上，然后用 2 kg 重的橡胶轮滚两次。

将每块钢板的一端放在 Instron Model 1122 拉伸试验机(Instron Corp., Canton, MA)的夹爪中，而将密封带的另一端粘贴在拉伸试验机的另一个夹爪上，以使基本上以 180° 的剥离角将带从帷帘上剥离。夹爪的移动速度为 30.48 cm/min，拉伸试验机记录从帷帘上剥离带层压物所需的压力。结果记录为牛顿/N/2.54 cm(N/2.54 cm)。起始粘合性测量是基于带在帷帘上停留时间短的事实，即实例带的粘贴和剥离之间没有长的停留时间(约少于 5 分钟)，而消毒还未进行。

对处理帷帘的消毒后粘合性

除试验前在 273°F(134 °C)对实例密封带蒸汽消毒 10 分钟重力循环(gravity cycle)，然后蒸汽干燥 3 分钟外，本试验使用初始粘合性试验相同的方法。所用的消毒器是 Barnstead Model GLS-10D (MDT Biologic Co., Rancho Dominguez, CA)。另外，剥离试验前，至少让层压物在室温下冷却 4 小时。结果记录为 N/2.54 cm。

20 静态剪切强度

按如下方法测试带的静态剪切强度。一块不锈钢板用双丙酮洗涤，用薄纸(KIM-WIPE)擦净，再用庚烷洗涤后，用薄纸擦净。庚烷洗涤再重复二次。将一段带子贴在 WraPel™ T 帷帘材料上，得到 2.54 cm × 2.54 cm 的粘合面积。带的其余部分悬在边缘，将其折成圈。将 2kg 重的滚轮沿带的纵向来回在粘合面积上滚动一次。将 250 g 的重物系在该圈上，所得的装置垂直悬挂于保持于 20 °C 和 50 % 相对湿度的室内。测量断裂的时间(带从不锈钢板上落下的时间)。

表 9

性质	
初始粘合性	229 N/2.54 cm
消毒后粘合性	88 N/2.54 cm
静态剪切强度	88 分
分散性	通过

30 虽然根据具体的实施方案对本发明作了说明，但应理解本发明可作进一步的修改。本权利要求书试图覆盖本技术领域中熟练技术人员认为是本发明的化学等

同物的所有变化形式。